THE SOCIETY OF PLASTICS ENGINEERS JOURNAL

EDITORIAL DEPT.
UNIVERSITY MICROFILMS, INC.
313 N. FIRST ST.
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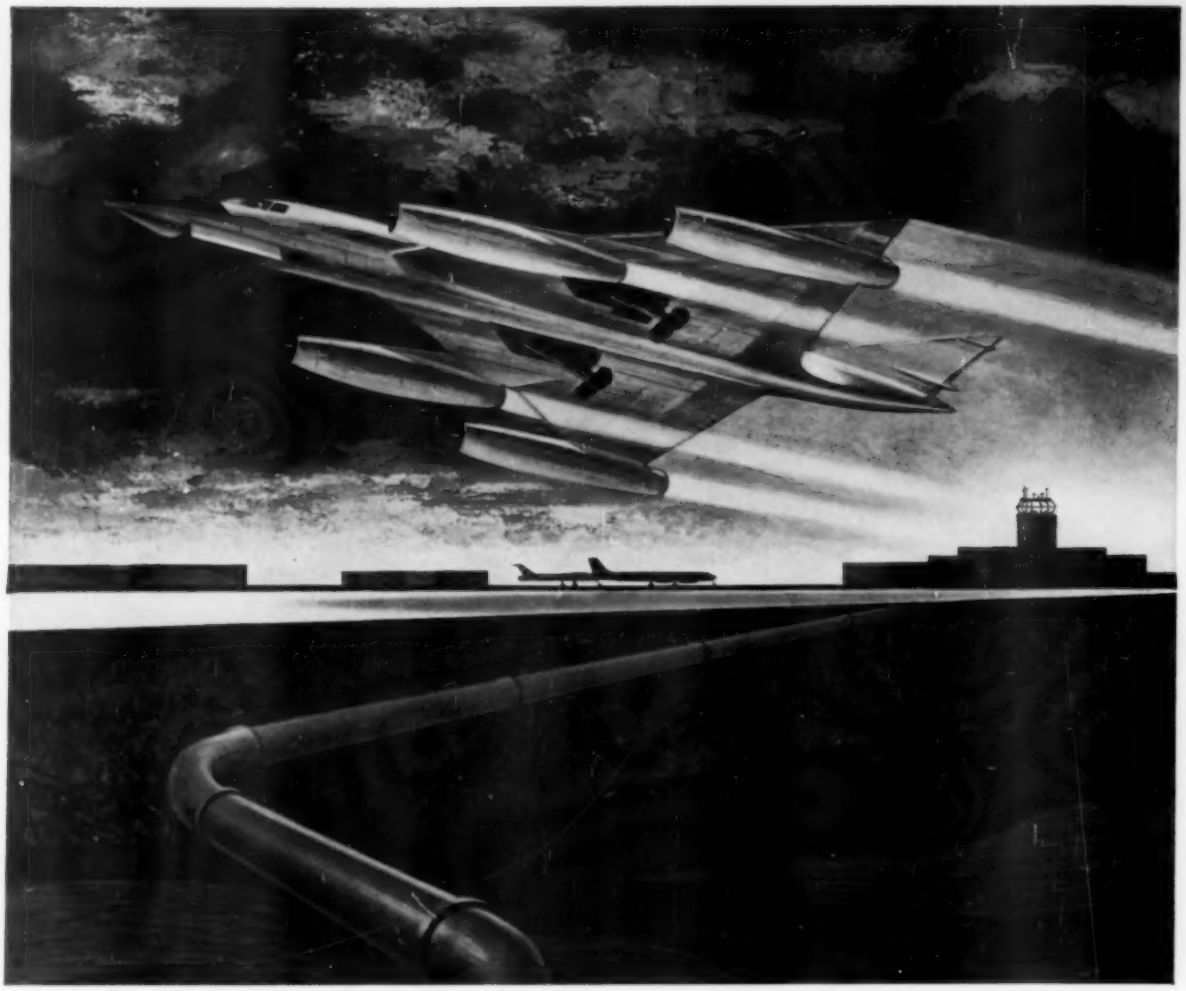
14-A

Users of resins for reinforced plastics applications get a lift from new method for controlling resin cure . . .
page 867

OCTOBER 1959 • VOL. 15, NO. 10



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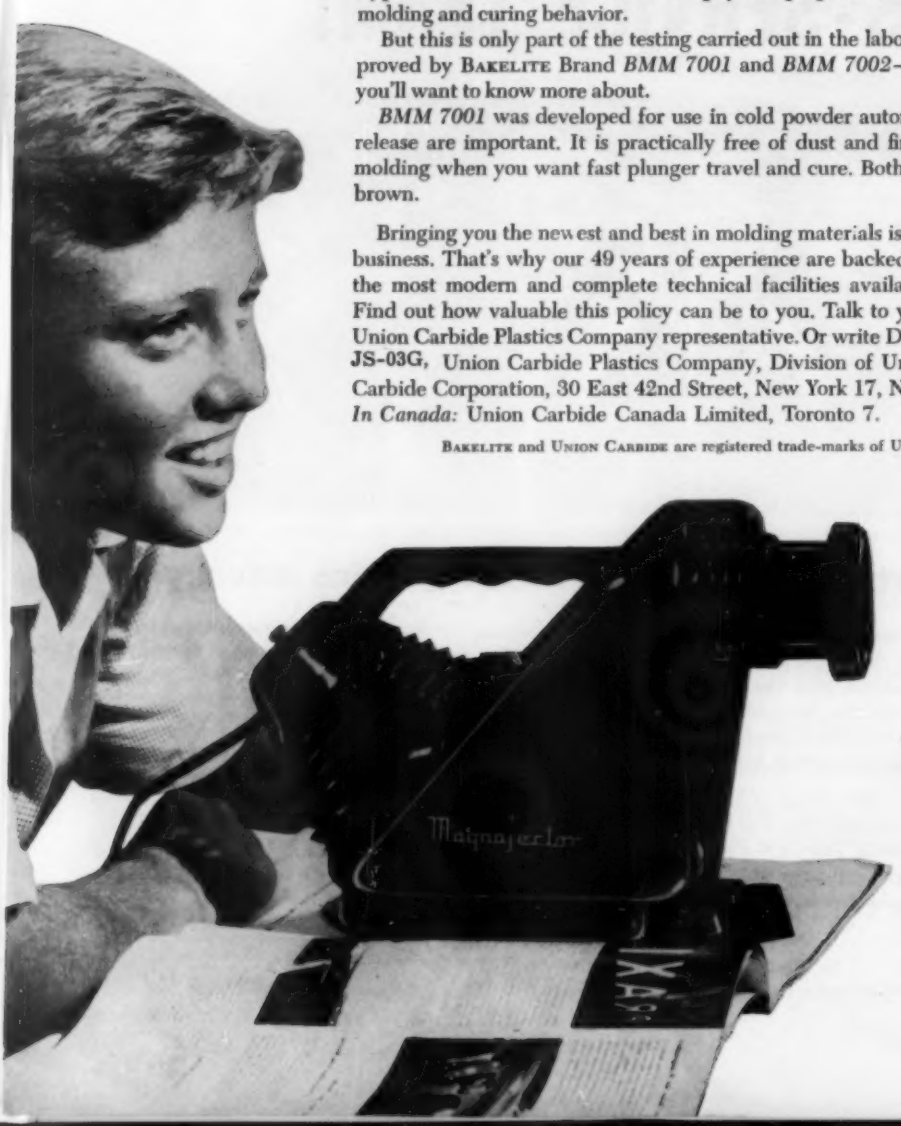

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The Society of Plastics

COVER

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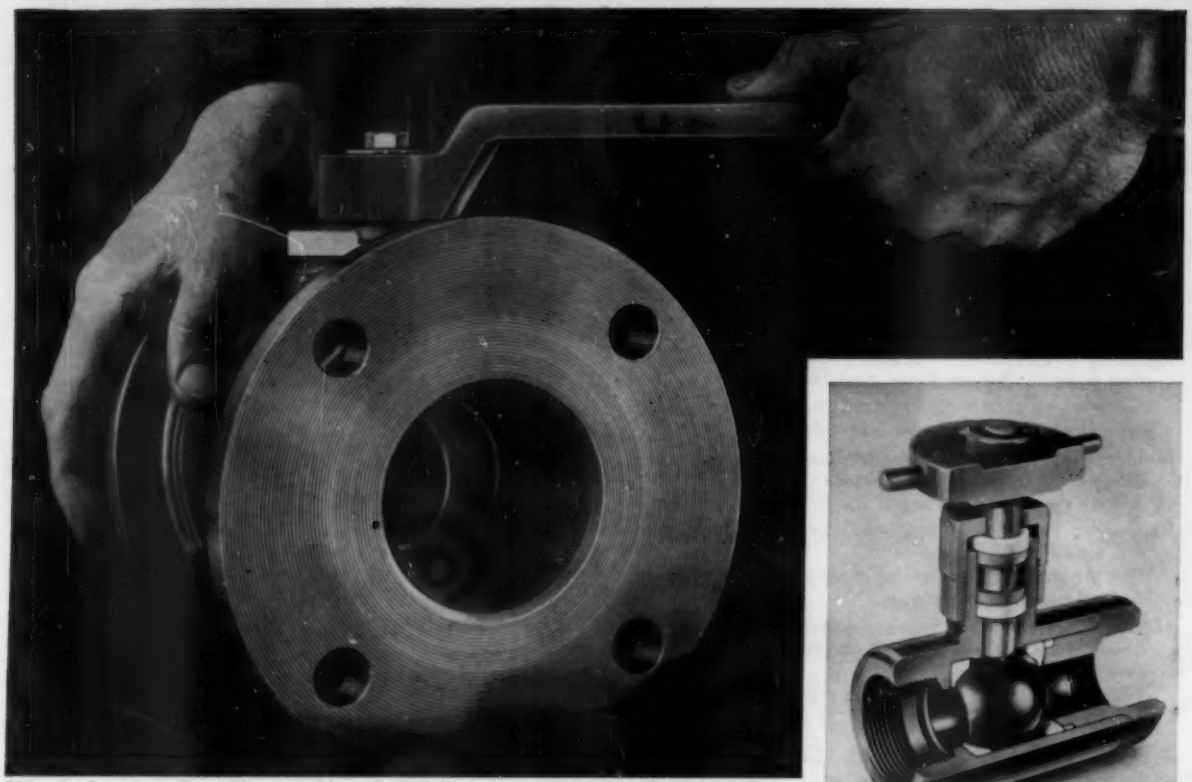

Valves by Jamesbury Corp., Worcester, Mass. Geon rigid vinyl parts molded by Mannesman-Easton Plastic Products, Inc., Easton, Pa. Valves in sizes from 1/2" to 4", shown in large photograph, are rated at 150 psi to 100 F. and at 50 psi to 130 F. They can be remote operated. B.F.Goodrich Chemical Company supplies the Geon rigid vinyl material only.

New ball valve of Geon helps piping systems fight corrosion

Thousands of test cycles with this new valve made from Geon rigid vinyl have proved the effectiveness of its seal, either at full pressure or vacuum. Since it is made entirely of Geon except for seats and seals, it offers pipe users another weapon for the battle against corrosion. Geon offers superior resistance to oils, acids, alkalies and most chemicals.

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U.S.I. POLYETHYLENE NEWS

A series of plastics and packaging developments by the makers of ETHOTHENE® polyethylene resins

Packaging Notes

Lightweight polyethylene-coated corrugated containers are being used to ship heavy electric typewriters.

Polyethylene was found to be the only material that did not damage the finish of the typewriters. A polyethylene-coated corrugated insert fits over the typewriter inside the container. This liner prevents damage to the typewriter, even if the box is overturned. The box cuts costs 30 percent, and volume 40 percent. Stacking strength is increased 40 percent.

A semi-rigid polyethylene bag inside a corrugated box is being used to package a liquid industrial deodorant. The containers, in quart and gallon sizes, pour easily and are convenient to handle.

This method of packaging reportedly eliminates the problems of freezing and bottle breakage and requires no deposit or return. The new containers require less transportation and storage space than glass or metal ones, providing lower handling costs.

Courtesy Canton Containers Inc.

In an unusually large packaging job, a locomotive was wrapped in a polyethylene film cover for export shipping. The cover was specially made to protect the locomotive from dirt, dust, and water spray during overseas shipment.

A new sealing machine for polyethylene bags reportedly provides substantial savings in packaging costs. The machine features an automatic sealing and cut-off device designed to save up to an inch of film length per bag. It eliminates conventional tabs and is claimed to save up to 15 percent in material costs.

Strong, flexible bags with many properties comparable to metal cans are being produced to package foods. These bags are made of aluminum foil laminated on both sides with polyethylene film, and coated on the outside with cellophane. They are impermeable to gas and moisture. The bags will heat seal, have high impact strength, and will stretch up to 28% (in machine direction) before rupture. They may be used for vacuum packaging, dehydrated food packaging, liquid food packaging. The packages can be boiled.

Growing Market for Polyethylene Film Seen in New Construction Applications

Film Makes Ideal Moisture Barrier for House and Highway Construction

A burgeoning market for heavy polyethylene film is developing out of new uses for the film in the construction industry. Three properties of polyethylene film have been chiefly responsible for its popularity with builders. It is an excellent moisture barrier. It is light-weight, yet strong. It is inexpensive.

Polyethylene sheeting in 4 mil thickness is placed on freshly laid concrete after a film of water is sprayed over entire area. Boards are placed over the joints and edges and left in place for at least 36 hours. Sheeting can be re-used for this purpose, or used as waterproofing under next slab in multi-dwelling construction.

Construction men are using polyethylene film as a moisture barrier under foundation slabs and concrete highway sections, between interior and exterior walls, and as a lining for crawl spaces. On one recent construction job, 70,000 square feet of 4 mil clear film was used under a concrete slab. Installation was facilitated by the light weight of the film — less than 20 pounds per thousand square feet. In house and highway construction, the same film used under concrete as a barrier, is frequently reused after first serving as a cover over other concrete.

On large construction jobs, polyethylene film has proved invaluable as an inexpensive covering for open, unfinished work. The film permits solar heat and light to enter, keeps out wind and dirt.

In house construction, film is being used for window and door flashing and as an under layer for shingle roofs. Film is being laid under shower stalls and in other parts of the house where moisture and water seepage is a problem.

The future of polyethylene in the building industry continues to be brightened by new applications. For example, "bubble houses" held up by air pressure show promise as economical shelters for construction work or for temporary storage. Polyethylene-coated plywood concrete forms that are self releasing have been suggested. New uses such as these promise to continue the spectacular growth which polyethylene has shown over the past few years.

Unsupported Polyethylene "Bubble" Houses Chickens

An air supported plastic shelter, used as a low-cost house for poultry research, has been built at a leading agricultural university.

No inside supports are used for the 10 by 20 foot shelter built with 4 mil polyethylene film. A one eighth HP motor keeps the quonset shaped shelter inflated. It is estimated the building will endure 60 mph winds and support a foot of snow. The success of the shelter suggests its use in meeting temporarily expanded farm storage and poultry shelter needs.

U.S.I. To Co-Sponsor Museum Packaging Show

U.S.I. will co-sponsor an international packaging show at the New York Museum of Modern Art from Sept. 9 to Nov. 9. Several hundred commercial and industrial packages selected from ten countries will be presented. They will range from wrapping materials to a 370 cubic foot container.

The exhibition is intended to re-examine and broaden the concept of packaging in relation to the great variety of products produced today. Outstanding examples of graphic and industrial designs will be shown with emphasis on imaginative use of new and old materials. Experimental and industrial packaging seldom seen by the public will also be exhibited along with well-known commercial applications.

New Slitter Is Faster, Reduces Trim Losses

A compact new machine is reported to shorten slitting time for polyethylene film and to reduce trim losses.

It normally takes 45 minutes to cut a 10,000 ft. roll of film, using a conventional slitter and winder. The new machine is reported to cut the same length of film — on the roll — in five minutes.

When the film is slit on the extruder, three to four inches of selvage material is usually lost. The new machine reduces wasted selvage to as little as $\frac{1}{16}$ of an inch. The new slitter eliminates the problem of fusing at the edge of the roll. The machine is said to cut film in gauges from $\frac{1}{10}$ to 10 mil, in any width up to 72 inches.

U.S.I.

Vol. IV, No. 5

POLYETHYLENE PROCESSING TIPS

IMPROVING EXTRUDER EFFICIENCY WITH PRESSURE GAUGE

Failure to determine the proper schedule for breaker plate and screen pack maintenance can be costly to polyethylene extruders. It can result in losses in production time and deterioration in the quality of the extruded product.

Changing a screen pack when it is not contaminated is an unnecessary waste of valuable production time. On the other hand, allowing the screen to become excessively contaminated poses serious production problems. The increased pressures that develop and the longer exposure of the polymer to heat can cause degradation and oxidation of the polyethylene. These undesirable changes in the resin's physical properties show up in off-quality end products. Excessive pressure, too, may damage the extrusion equipment.

Pressure Gauge Guides Operator

These difficulties can be reduced by installing a pressure gauge in the extruder cylinder wall between the screw and the breaker plate, as shown in the drawing, Fig. 1. The gauge continuously monitors breaker plate condition and indicates to the operator when it is time for screen pack changes.

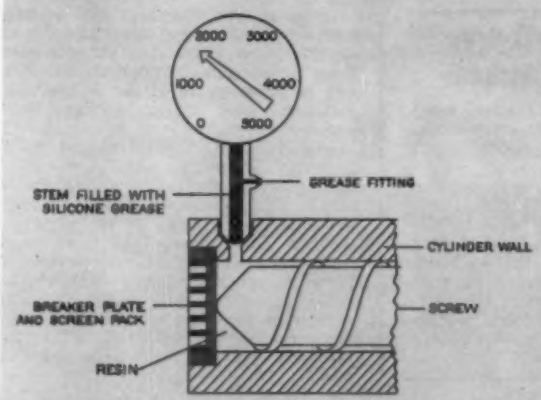

Fig. 1. Diagram of installation of pressure gauge on an extruder

As the accompanying chart, Fig. 2, shows, pressure in the cylinder increases as the area of openings in the screen pack decreases, that is, as screen plugging increases. Pressure, of course, is dependent on operating temperature, screw temperature, and screw speed as well. In practice, the operator first determines operating conditions and screen pack arrangements that produce the best results with a given product, using the pressure gauge as an aid in such preliminary runs.

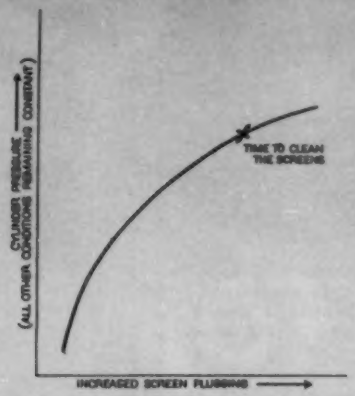

Fig. 2. Cylinder pressure increases with increasing screen plugging

Once operating conditions are established, pressure can be correlated with contamination in the screen pack and breaker plate by experience. The build-up of pressure beyond a given point is the signal that the screen pack needs changing.

How Gauge Operates

The bleeder-type bourdon tube gauge is the pressure-measuring device most commonly used in the extrusion industry. The gauge can be installed on the top or bottom or at any angle on the cylinder. The tube running from the gauge to the cylinder is filled with silicone grease introduced through a grease fitting.

Gauge Maintenance

The tube, or stem, must be kept filled with silicone grease, or else polyethylene resin will be forced into it from the cylinder. If this happens, the resin will solidify, causing inaccurate pressure readings on the gauge. Applying grease through the fitting once every 24 hours of extruder operation should be sufficient. It is best to add the grease between production runs so that the small amount forced into the resin can be easily purged.

If the stem becomes plugged with resin, it can be cleaned quickly. The operator simply removes the stem, heats it, and purges it of resin by forcing silicone grease through it.

Technical Assistance from U.S.I.

Most troubles associated with screen pack and breaker plate maintenance should disappear with the installation of a pressure gauge. But if you have special problems, ask U.S.I. for technical assistance. Our technical service engineers will be glad to work with you in solving them.

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THE EDITOR'S *Notebook*

Plastics International

We are sitting in our office and the phone rings. It's an SPE member just returned from Moscow. He's calling to offer a written report on plastics research in Russia. Another member, and Past President of the Society, Jerome Formo, recently returned from Poland where he observed the latest advances in Polish plastics research. Report of his visit, incidentally, is on page 858 of this issue. Still another member and officer of SPE, Jules Lindau, III, Second Vice President, recently returned from visiting plastics plants in France and England. And, about the time you are reading this editorial, a planeload of SPE members will be leaving New York's Idlewild Airport for the Dusseldorf Plastics Exhibition in Dusseldorf, which opens the morning of October 17, 1959. Undoubtedly, other SPE members are planning to attend the International Plastics Exhibition in Oslo, Norway, on May 20-31, 1960.

All this activity in international travel is significant. It points up the keen interest of American plastics engineers and scientists in what their counterparts are doing overseas. And so far, comment has been to the effect that the caliber of the work being done abroad in the plastics field is high.

While scientific curiosity is enough to justify wanting to know what's going on in the other fellow's laboratory and plant, there's more to it than that. The rapid growth of industrial capacity in other countries poses the problem of our ability to compete technically and economically in the world market place. Too, there is already a well-established trend towards the producers and users of plastics products "going international".

But for companies in the plastics field, why go international at all? A large part of the answer lies with precedence. Plastics have always had strong application in consumer goods—housewares, toys, and more recently in boats. Today in most parts of the world, the people are displaying an eagerness for such consumer products. More important, the governments of those countries now recognize that such consumer items are a necessity and have just turned the valve on, so to speak. Completing the picture, there is enough prosperity in many foreign countries now to support purchases of consumer goods.

Another reason producing abroad can be wisdom is the fact that international status can give a company lower wage costs and better tax rates. Just recently, tax aid programs have been instituted this year by France, Belgium, Luxembourg, and Holland. Italy has for some years helped industries locate in the underdeveloped Southern section of Italy.

There can be no doubt that the increased travel of our technical representatives overseas will result in more companies, both producers and users, with substantial international interests.

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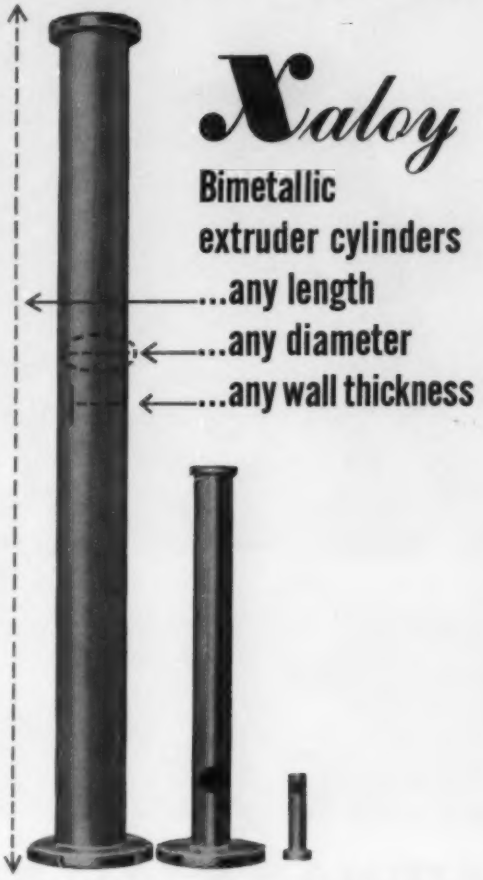

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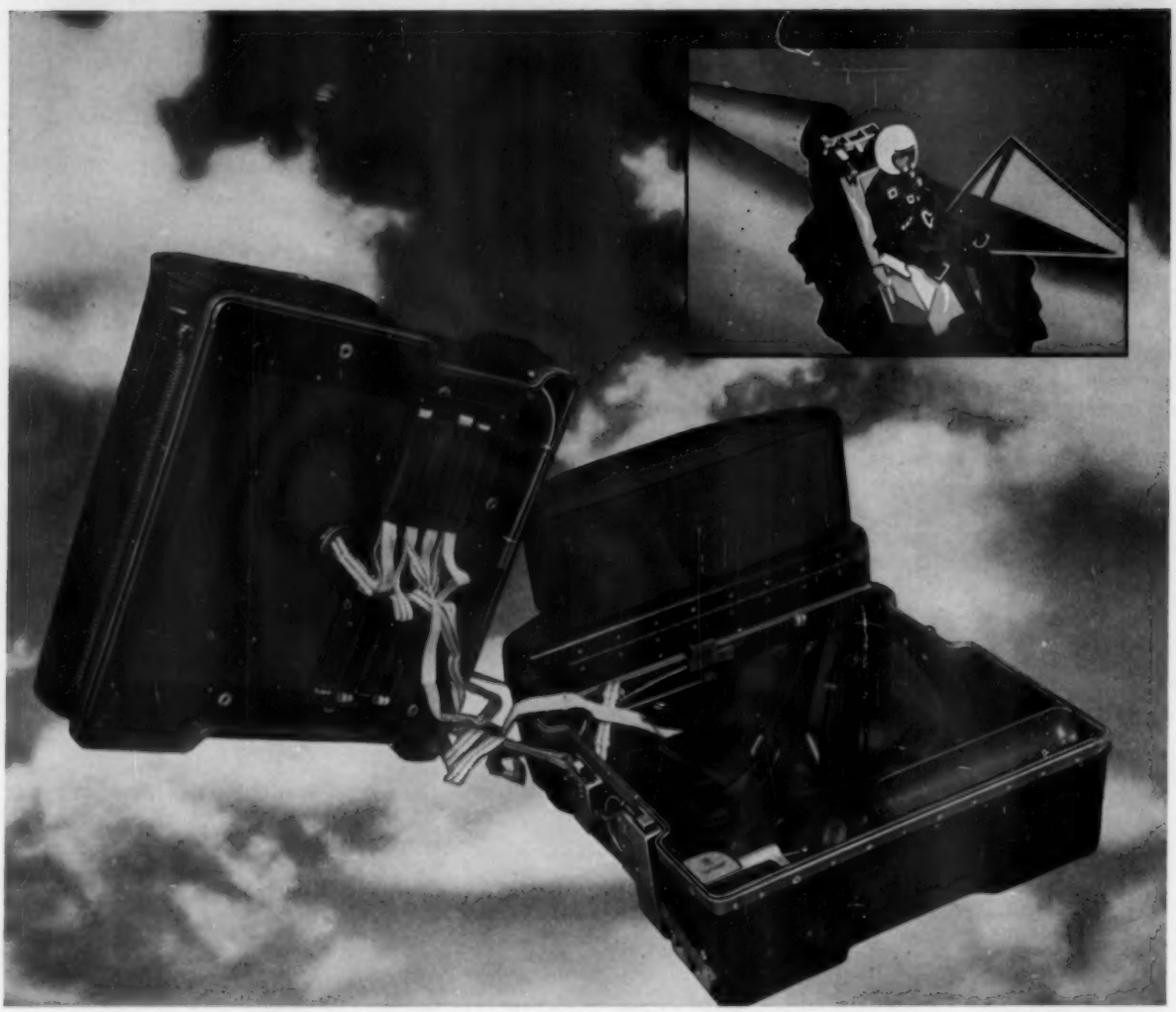

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And even more interesting: how the survival kit can withstand the sudden shock of a rocket-fired ejection at fighter-plane speeds, and the impact of a parachute landing in rough terrain is a lesson in the advantages of reinforced polyester molding by the Plastics Division of General American Transportation Corporation.

Various materials were tried, but lacked the high impact strength-to-weight ratio and the necessary resistance to dents, corrosion, and fungus growths of the reinforced moldings. Also important is the economy of reinforced plastic molding. Complex configurations, holes, rounded edges, even color, can be molded in the part, reducing the number of costly operations.

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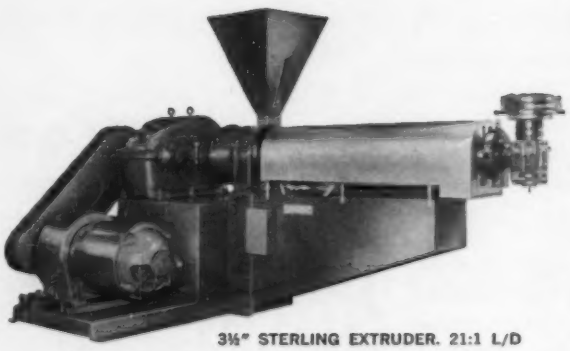

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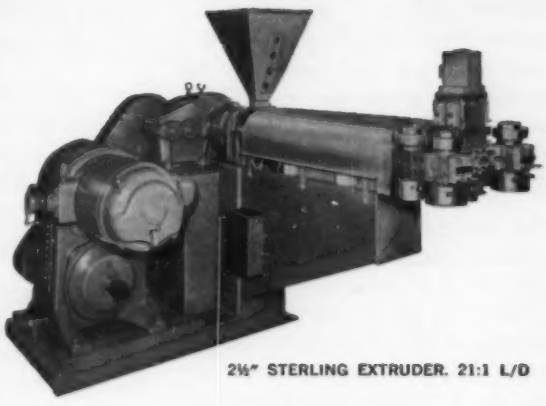

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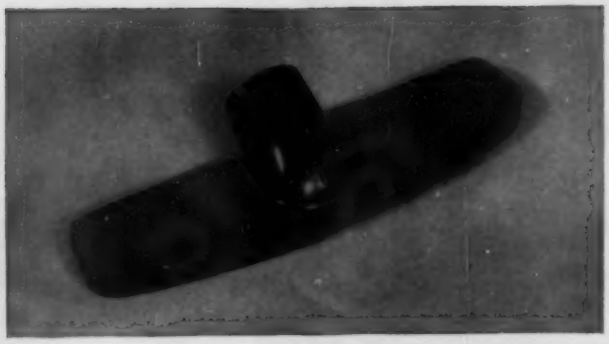

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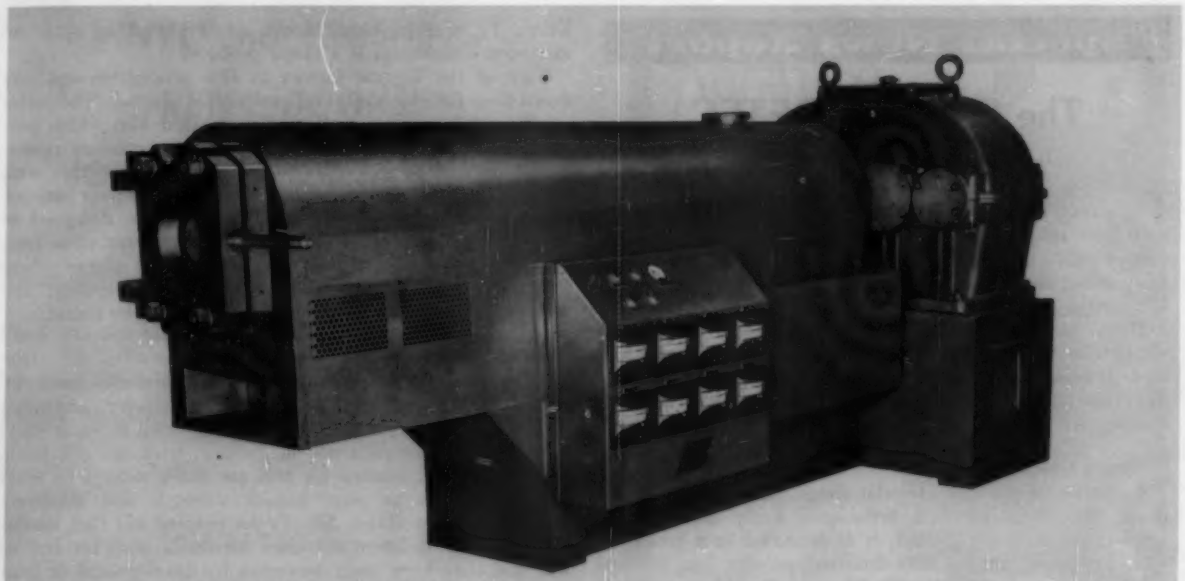

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Farrel's line of extruding machines for the plastics industry has been expanded to include small Inductomatic extruders in a range of sizes from 2½" to 6". These are used for the extrusion of thermoplastic products such as tubing, clothesline, pipe, hose, gaskets, wire coating, blown or flat film, sheet, compounding, etc.

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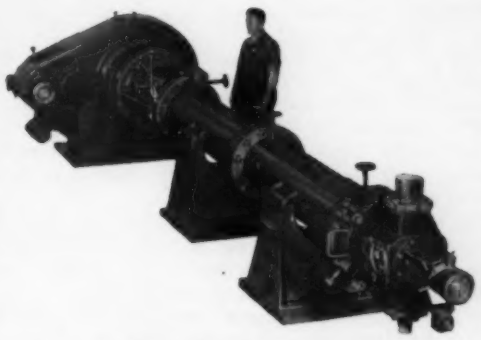

8½" UNDERWATER PELLETIZING EXTRUDER

Among the wide variety of large processing extruders built by Farrel to individual requirements is this cold-feed machine. It was designed to rework cold granular polyethylene with antioxidants and, with or without coloring matter, force the material through a straining screen, and pelletize it in an underwater head at a rate of 1600 pounds per hour.

Special News Report

The Cleveland RETEC

Jesse H. Day, *Ohio University*

The great versatility of the vinyl resins and the tremendous scope and variety of their uses was clearly demonstrated by the range of papers presented at the Cleveland Retec on Vinyls, held October 7th at the Cleveland Engineering Center.

Blow extrusion of PVC film and sheet, casting of vinyl film, rigid vinyl sheet, PVC solutions for coatings and plastisol and fluidized bed vinyl metal coatings provided the subject matter for six papers presented during the day.

Fluidized Coatings

Large metal parts of irregular shape can be vinyl coated using the Fluidized Bed technique. Fully compounded vinyl material, finely ground, is air-levitated in a container with a porous bottom. The fluidized powder has the appearance of a boiling liquid. The metal to be coated is heated above the fusion temperature of the vinyl and then immersed for a few seconds in the fluidized powdered vinyl. The powder fuses to the surface of the metal and provides a continuous coating. Thin metal pieces may require a short postheat to provide a high gloss.

The fluidized bed process was described by *J. R. Russell* of Steere Enterprises. He added that the method was particularly useful when the part cannot be coated well by plastisol dipping due to dripping or air entrapment. Expanded metal grille work can easily be coated by this method, and has the advantage of covering equally well any protruding sharp edges.

Vinyls Fight Corrosion

Industry's five and a half billion dollar annual loss to corrosion is being fought effectively with vinyl and modified vinyl coatings, according to *C. W. Wright* of the Glidden company. Formulations and methods of application depend on the particular corrosion problem. Where severe chemical resistance is needed straight vinyl coatings are used, using a primer coat to give the vinyl film firm adhesion. Wright quoted Joseph Bigos of U. S. Steel on the fact that vinyl painting of galvanized steel is greatly effective in thin films if some zinc is incorporated.

The great variety of uses for vinyl protective and decorative coatings is illustrated by its use in metal food and drink containers, paper coatings, fabric coatings and industrial protective paints. Maximum protection is achieved with straight vinyl, but poor metal adhesion requires a primer. Vinyl modified alkyds are used for one layer application, and plasticizers and stabilizers improve weather resistance, flexibility and heat stability.

Cost factors can be adjusted by judicious planning; straight vinyl requires expensive solvents, but alkyd modified vinyls can use solvents costing less than a third as much.

Mr. Wright added that the number of coating formulations is so great that the consultation and understanding of the paint formulator is needed to assure the best results.

Blow Extrusion of PVC

Blow extrusion of PVC film and sheet is usually complicated by the fact that the end-use properties needed dictate the use of plasticizers, stabilizers, and lubricants which are often not the materials best suited to make the extrusion process work easily and well. *Philip H. Rhodes* of Philip H. Rhodes Associates pointed out that the selection of formulations to meet customer needs while providing optimum extrusion compounds is a major problem.

Two of the biggest factors in film production cost are down-time for clean-up, and production wasted. The latter can be minimized by running the greatest film width possible, and slitting to smaller widths in a secondary operation, but the economic problems of downtime, together with the practical problem of getting a smoothly running process, depend heavily on die design. Dies must be designed to provide minimum variation in film thickness, and close temperature control combined with avoidance of places where plastic can lodge to begin decomposition or burning. The complete solution to the die problem is yet to be found.

Coming: Blow Molded PVC Containers

Tremendous potential uses for vinyl products made by blow molding are being overlooked by industry, according to *Don. M. Taylor,* consultant. Although such items as vinyl bottles are in common use in Europe, they are not made here. Suggested reasons for this are naive ratings of vinyl properties such as poor impact strength and relatively high gas permeabilities. Mr. Taylor pointed out that similar problems are inherent in other materials used for bottles but that they have been overcome by development of liner coatings. Vinyls provide solution coatings which have excellent adherence to the prime vinyl container and provide permeability values unmatched by other thermoplastics.

Development of blow molding techniques for the vinyls will find potential markets of 125 million pounds for containers each year, with similarly large outlets for such items as doll parts, footwear, detergent containers, chemical containers, and bottles of many kinds.

Environmental Tests—On a Mill

Heat stability tests on vinyl compounds for rigid sheet are more reliably run on a laboratory mill than by the usual oven tests. Seiberling milled compounds on a small laboratory mill at 370°F—or 320°F for copolymer—and took samples at five minute intervals until discoloration. Formulations showing no appreciable discoloration after 30 minutes prove satisfactory in production. Since processing of rigid vinyl must be done at temperatures near the decomposition point, mixing is best done in a Banbury to avoid the high heat build up during the long time required to mix on the mill. Because stock temperature approaches 400°F in the nip of the rolls, calendaring should be done on a calendar with the fewest number of rolls.

Expanding markets for rigid vinyl are due to continued improvements in material and in the skill of experience. The compounder has three general types of resin to fulfill a variety of property needs; the pure vinyl and vinyl which incorporates acrylic, chlorinated polyethylene or ABS for high impact strength. Both are outstanding in resistance to acids and bases, hence their use in duct work, plating tanks and pipe fittings. A third resin is the copolymer of vinyl chloride with vinyl acetate which is easily heat formed and readily accepts printing inks, for use in skin packaging, luggage and light fixtures.

Sheet extrusion of rigid vinyl is a comer, but most sheet forming is presently done by calendaring. Calendaring is limited to sheet thickness of about 0.035 inch and thicker sheets are made by lamination.

Cast Vinyl Films

The largest potential use for cast vinyl films will probably be for coated steel sheet. Aside from this, there are no large new markets in sight, according to *D. S. Threlkeld* of the Clopay Corp. Present uses include laminates to woven fabrics for adhesive bandage, adhesive tapes, baby pants and automobile seat covers, and coatings on paper for table tops, luggage and box covers.

VINYL and FINISHING RETEC BOOKS JOIN SPE PUBLICATIONS

Announcing

Vinyl Plastics—Cleveland Section, 7 papers. $2.50, members; $3.75, non-members. (See program, SPE Journal, August, 1959 issue, p. 745).

Plastics Finishing—Buffalo Section, 4 papers, $2.00, members; $3.00, non-members. (See program, SPE Journal, September, 1959 issue, p. 823).

Other 1959 RETEC Preprint Books

★ **Encapsulation, Printed Circuits, and Fluidized Bed Processes**—Northern Indiana Section, 15 of 23 papers presented. $3.00, members; $4.50, non-members. (See program SPE Journal, April, 1959, p. 319).

★ **Plastics in the Automotive Industry**—Detroit Section, 4 papers, $2.00, members; $3.00 non-members. (See program SPE Journal, May, 1959, p. 443).

★ **A Designer's Look at Reinforced Plastics** from the Pleasure Boat, Aircraft and Missile Industries Viewpoint—North Texas Section, 6 papers. $2.00, members; $3.00, non-members. (See program SPE Journal, April, 1959, p. 324).

★ **Plastics in the Metals Industries**—Pittsburgh Section, 10 papers. $3.00, members; $4.50, non-members. (See program SPE Journal, April, 1959, p. 324).

SPE PLASTICS ENGINEERING SERIES

Vol. II—Processing of Thermoplastic Materials, 705 pages. $14.40, members; $18.00, non-members.

Vol. I—Quality Control for Plastics Engineers. $3.98, members; $4.95, non-members.

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Vol. V, 1959, New York City, 96 papers. $7.50, members; $12.50, non-members. Single preprints of individual papers, Vol. V, while they last, $0.25 each, members; $0.40 each, non-members.

Vol. IV, 1958, Detroit, 94 papers. $5.00, members; $7.50, non-members.

Vol. III, 1957, St. Louis, 60 papers. $5.00, members; $7.50, non-members.

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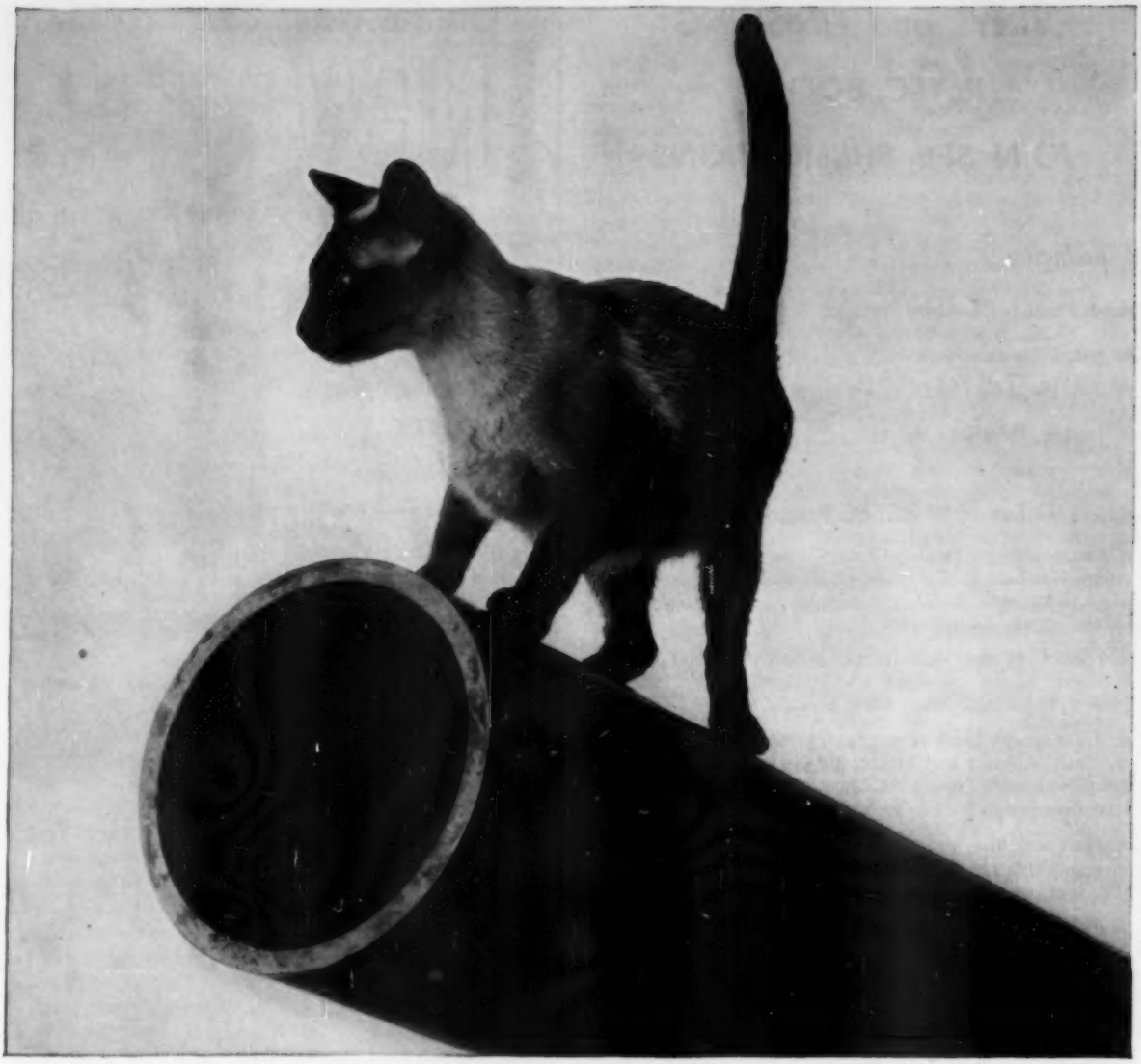

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Plastics Research in Poland

In Poland today, one can see heroic efforts being made to literally "boot-strap" the Polish plastics industry into existence. And the efforts have not been without visible results.

Recently, I had the privilege of representing the U.S.A. on a Trade Mission Team which toured Poland, visiting several plants and meeting with hundreds of Polish technical people. I visited the Plastics Research Institute in Warsaw, which is the research center for all Polish plastics. Ing. Konstanty Leidler, Director, Plastics Section, Ministry of Chemical Industry, is directly responsible for the development and growth of our industry in his country. He is the driving force—encouraging, challenging, needling his people into a more rapid development of the industry. If one considers that essentially all of this has been done since 1951, it must be granted that good progress has been made.

In 1949, about 790 metric tons of phenolic resins and 613 metric tons of cellulosics were produced. Other resins were almost unknown. In 1958, phenolics had grown to 11,400 tons, amino resins had entered and developed to 5,100 tons and polyvinyl chloride had become important to the extent of 4,200 tons. Resins which helped increase plastics production included also caprolactam nylon, alkyds and polystyrene.

Since all research necessary to develop these materials was accomplished at the Plastics Research Institute, it was especially interesting to visit this facility which is under the direct supervision of Dr. Marcus Wajnryb. The Institut Tworzyw Sztucznych occupies a building in the complex housing the research facilities of the chemical industry, and utilizes the common library. Outstanding in the Research Library is the collection of Eastern literature, principally Russian and Japanese. About 16,000 volumes in Russian, Japanese, English, French, German and Polish make up the technical collection. In addition to Beilstein and Chemical Abstracts, Polish researchers have available the Russian Abstracts as well. A new publication, Polish Technical Abstracts, was shown us as an indication of the serious nature of their work. The Plastics Institute also publishes its own technical journal but some publication difficulties are apparently being encountered because it is now several months behind schedule.

The Chemical Ministry has allocated about 1% of the value of plastics production to plastics research. This enables the Institute to employ about 170 workers. The work is divided into ten departments, each specializing in certain activities such as testing, mechanical, chemical, electrical, and application work such as vinyl films, floor tiles, etc. Twenty senior laboratory heads each have one or more small lab units of 3-5 workers under a director (PhD). About 70 of the staff have degrees.

As the budget is increased, new researchers are expected to be drawn from out in the industry since no technical schools provide the specialized training desired. Other plastics laboratories of a very small nature exist in the material manufacturing plants. These are primarily devoted to control and the improvement of the specific products manufactured.

Research Objectives

Plastics research in Poland was undertaken in 1951 and was aimed specifically at materials which could be put into production quickly. Thus, caprolactam nylon known as Steelon came into existence and it is now being manufactured at a large modern plant in Gorzow.

The present capacity of the Steelon plant is 3,500 metric tons per year with plans to raise this to 12,000 tons by 1965. The entire technical staff at Gorzow is Polish and Henryk Welter, the Director, is very proud of the fact that Steelon is entirely Polish from research through pilot plant and into production. He is also proud of the Polish designed and built reaction vessels and auxiliary equipment used in his plant. At present, most of the production is channeled into textile fibers with only a small proportion being diverted to plastics fabricators.

The Gorzow plant supplies about one-fourth of its own raw material, caprolactam, by conversion of phenol and depends on other Polish plants for the balance. A small triacetate film pilot plant is also operated here which is aimed at supplying needs of the new packaging industry.

Emphasis on Analytical Procedures

Currently, the emphasis at the Plastics Institute is heavy on methods of identifying and controlling impurities in the production of polyethylene and polystyrene. Satisfactory methods of manufacturing polyethylene have not been developed even though almost every available research tool including the electron microscope, ultra violet, x-ray, and infra red analysis is being used. Old and inadequate equipment is often the chief problem of the dedicated researchers hard at work on these problems. However, notable exceptions are evident, especially in physical testing equipment. Through membership in the International Standards Organization, the Poles are maintaining contacts with other technical people. This may account for the fact that the best equipment in the Institute seems to be of this type.

The trace studies being made also include standard spectro-photometric means, both emission and flame photometry, as well as x-ray spectrophotometry. Special studies are under way to follow the disappearance of crystal structure in polyethylene with increase in temperature.

Poland's major plastics import is PVC, most of which comes from the Eastern bloc. This vinyl is used largely in unsupported film for consumer goods. Considerable research effort is underway evaluating stabilizer systems. Thus far, soda stabilized emulsion type PVC seems best to them. They have also found that organic stannic stabilizers are best for thermal stability, but lack in adding light stability. Epoxy with zinc naphthenate has thus far given them the best Atlas weatherometer resistance.

In another section of the Institute's laboratory, basic studies are being performed with various phenolic bodies with the end view of utilization in higher phenol resins. Paper chromatographic techniques are used here. This laboratory section has published the synthesis of such products as 3-hydroxy methyl phenol. Epoxy resin studies currently are limited to the field of electrical insulating varnishes. Durable and very adhesive films have been prepared and are tested with the Erichsen rounded cone test. DIN German standards are generally used in this laboratory.

Undescribed work utilizing a pilot plant for the production of silicone resins had recently come to a halt and the equipment was being dismantled for shipment to some production site.

The impression most forcibly received during this visit was that of a dedicated group of scientists and technical workers thoroughly in love with their work. This was very evident in many of the laboratories and must account, at least in part, for the results thus far obtained. Certainly facilities and modern instruments cannot claim a major portion of such credit, since they are not generally available except in certain cases.

Poland's big problem is selection of research projects. Since limited facilities and maximum output are both required, the selection of projects which will result in almost immediate utilization seems to be the goal. Considerable difference of opinion exists within the Polish ranks as to whether the present plan best meets the ultimate goal.

One thing is certain. The Polish technical and scientific people will give a good account of themselves and will continue to strive hard to develop new materials for production in Poland. They know much better than we do what it has meant to do without many consumer goods we would consider essential, and they fully intend to do something to correct the situation.

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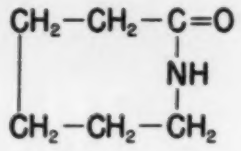

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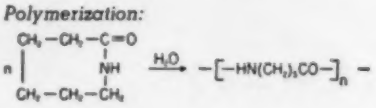

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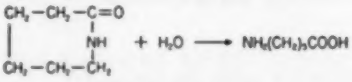
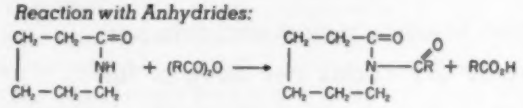
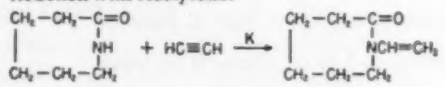


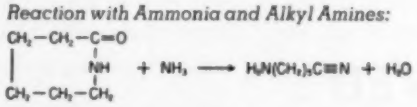
Reaction with Anhydrides:



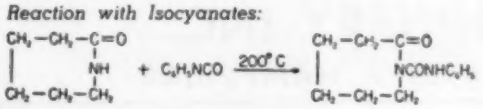
Reaction with Acetylene:



Reaction with Ammonia and Alkyl Amines:



Reaction with Isocyanates:



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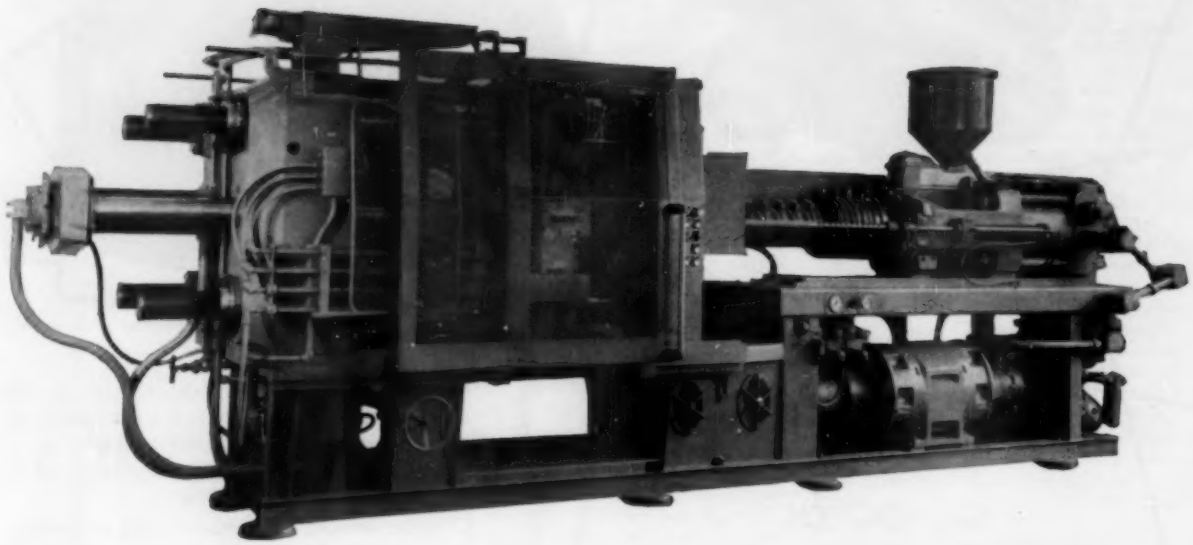

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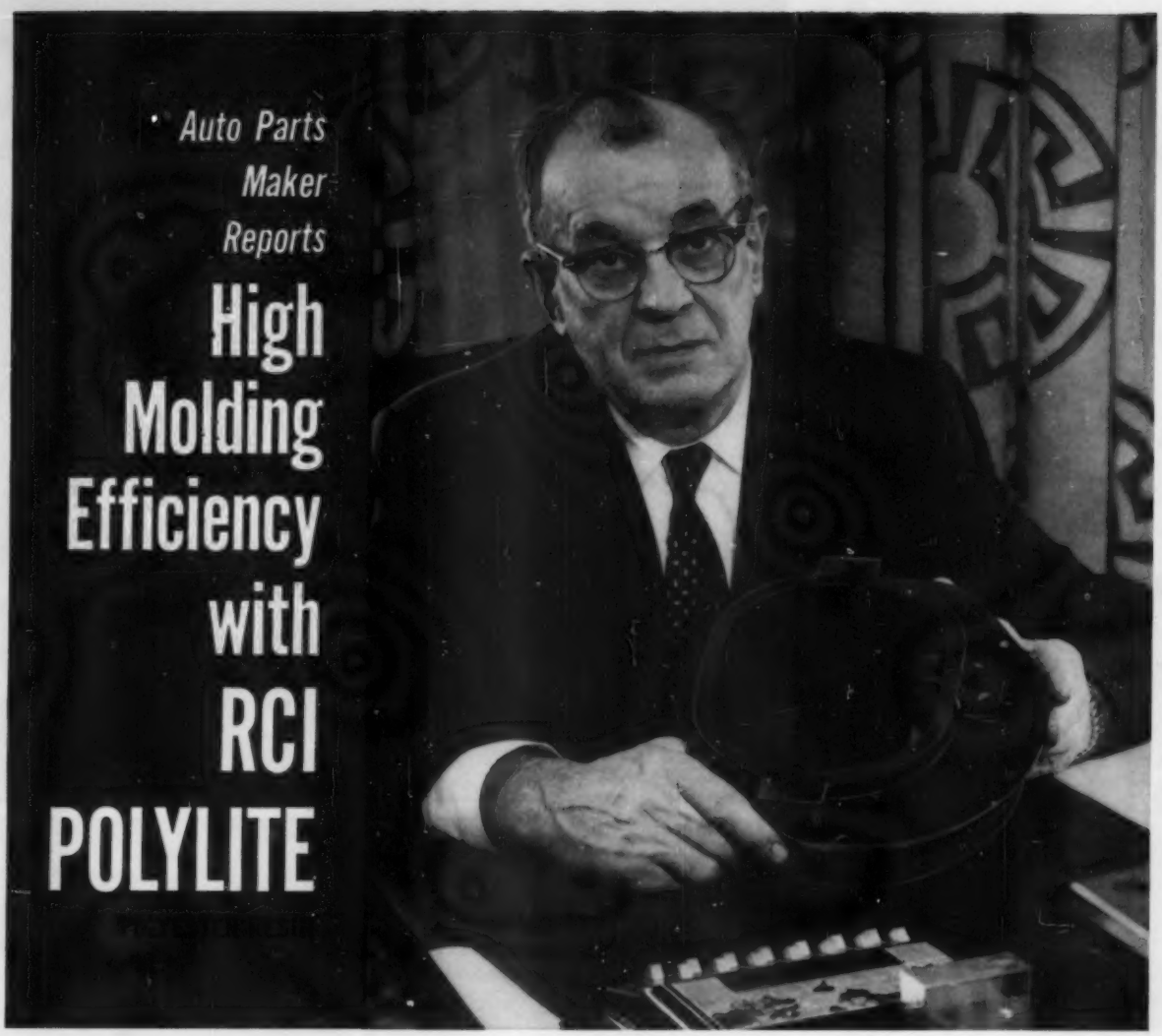

Cure Time Reduced to 42 Seconds

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"POLYLITE gives us about four times the curing speed obtained with other types of molding material. Press time with POLYLITE is only 38 to 42 seconds as compared to three minutes with other materials," continues Mr. Barnum.

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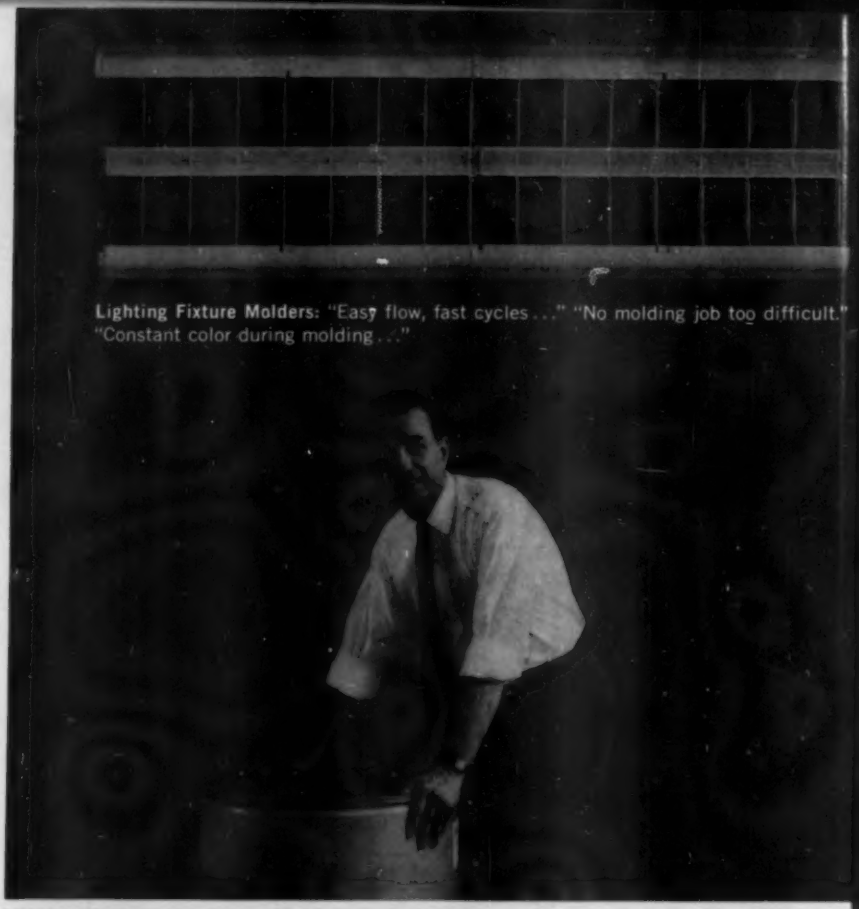
WHAT THEY'RE SAYING ABOUT LUSTREX* PERMA TONE STYRENE



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Lighting Fixture Manufacturers: "2 to 3 times more resistant to yellowing . . ." "Exceeds IES–NEMA–SPI specs for light stabilized styrene." "Excellent crystal clarity . . . unlimited color possibilities."

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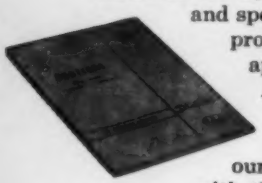

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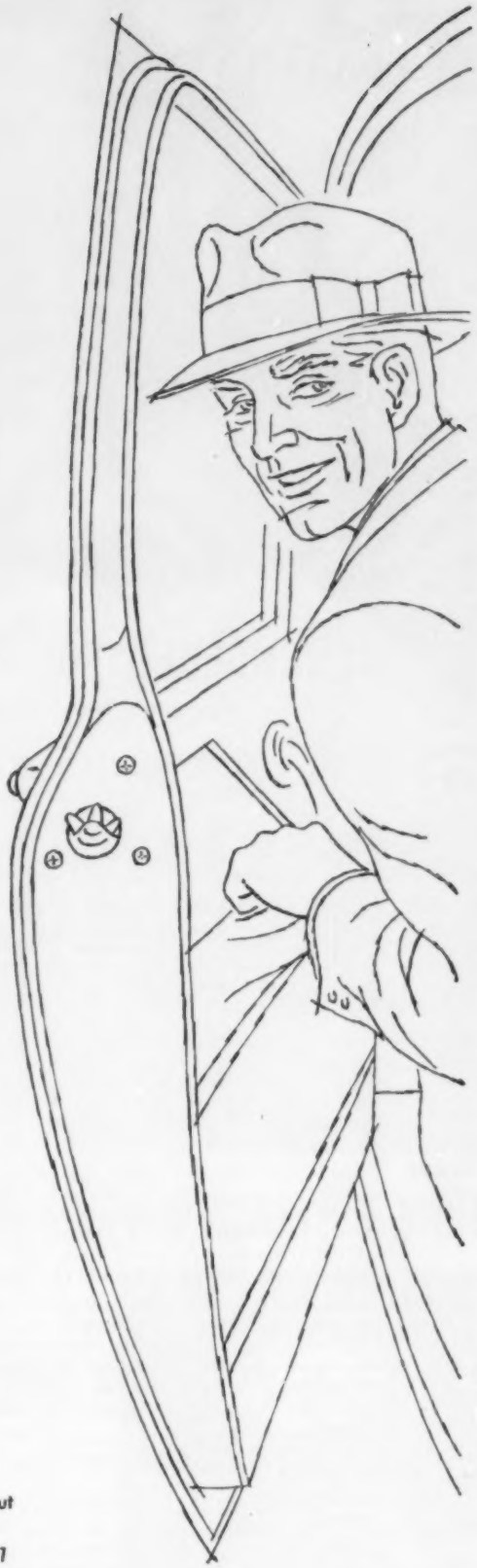

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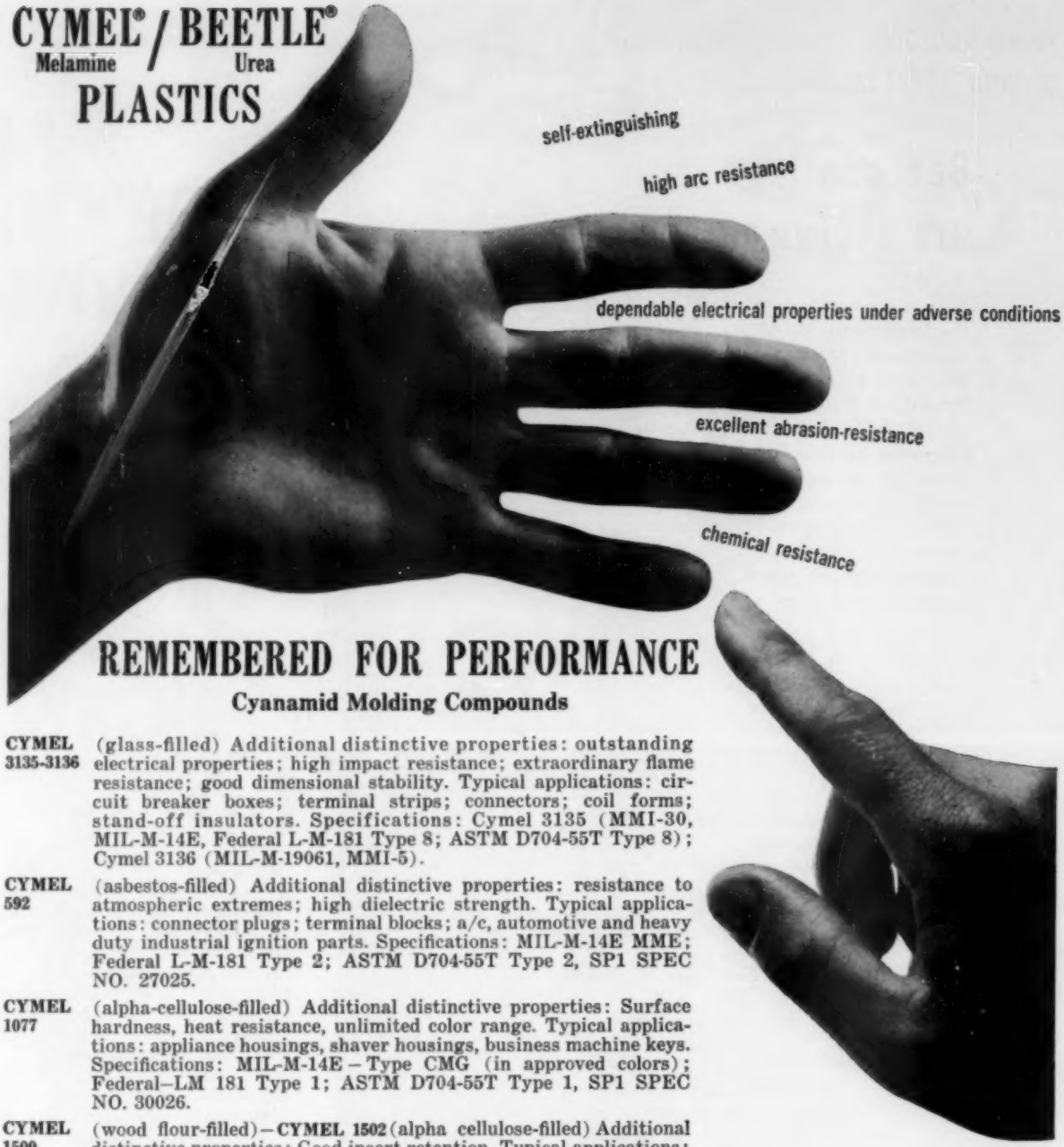

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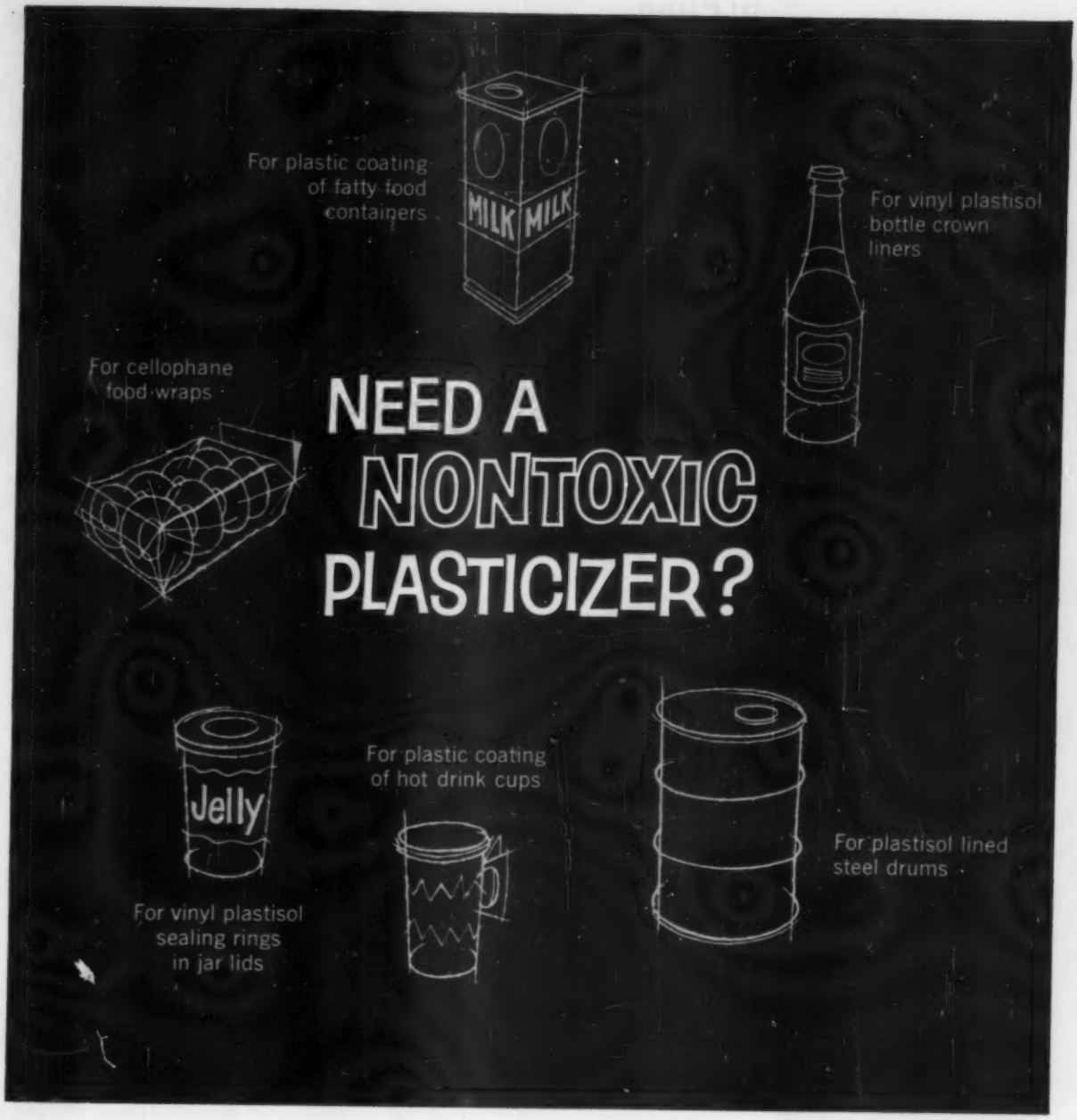
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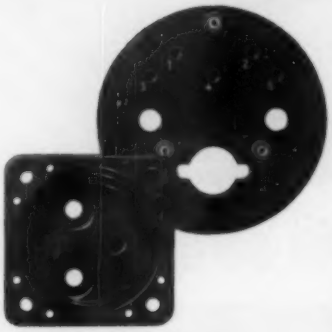

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SPE Journal

Volume 15, Number 10, October, 1959

James M. Smith[1] and
Robert B. Bennett[2]

An Easy Way to Control Cure of Polyester Resins

This article gives a method for controlling the cure of polyester resins. The method is based on the use of two types of graphs, with detailed steps given on how to obtain data for the construction of these graphs. More important: These steps may be used to obtain similar information for any other similar resin.

Since World War II, when polyester-glass laminates were developed which could be cured without pressure, many small as well as large companies have been using them to manufacture boats, swimming pools, radar domes, and other large plastic structures. Losses of the expensive resins and glass cloth, often have resulted from overcures and undercures caused by changes in atmospheric temperature and humidity.

The first part of this article presents the practical side of the control of the cure of these resins, by means of two types of graphs. These graphs could be furnished by the manufacturer or modified by the operator to obtain optimum and controlled cures in spite of wide variations in the seven variables which must be considered. These variables are: Per cent catalyst, per cent accelerator, room temperature, relative humidity, and the particular choice of resin, catalyst, and accelerator or their solutions.

Although larger companies often augment contact pressure by using vacuum or air-operated bag or mold pressure, with or without controlled heat, the same types of graphs could be constructed to suit the conditions of manufacture. In this case, the humidity variable could be replaced by pressure or strength relationships.

The steps which may be taken to obtain better control of the setting or gel time are given below, and are followed by a detailed procedure to be used to obtain similar information for any other similar resin.

[1] Now employed by the American Oil Co., Texas City, Texas.
[2] Professor of Chemical Engineering, University of Florida, Gainesville, Fla.

Application

Only two types of graphs are needed for any one selection of a resin, a catalyst, and an accelerator.

The first type of graph (Fig. 1) plots air temperature against a range of accelerator/catalyst ratios on semilogarithmic paper. Suppose that the operator has been using a combination of resin, cataylst, and accelerator recommended by the resin supplier and wishes to allow for a change in room temperature and humidity so that he can obtain the same gel time experienced on a previous laminated structure. In Figure 1 the operator has chosen a gel

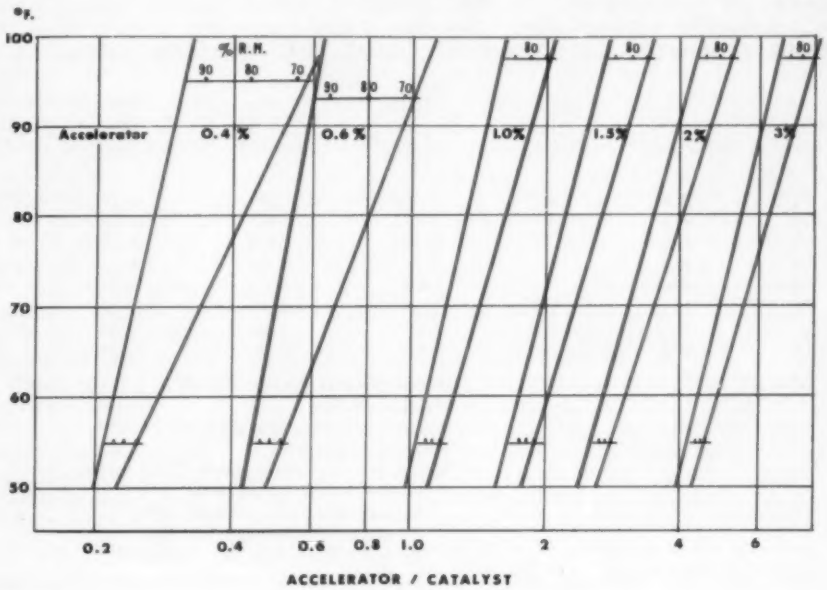

Figure 1. By using a combination of accelerator concentration, relative humidity, and temperature, this graph can be used to control the cure of polyester resins.

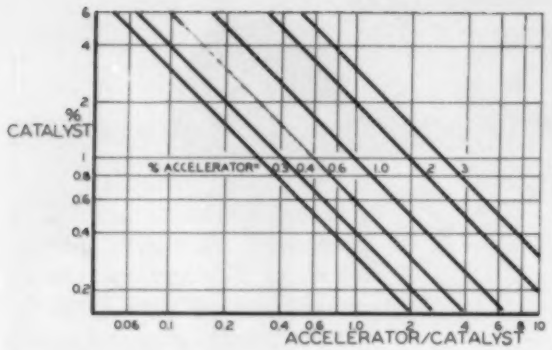

Figure 2. Graph shows an arithmetical relationship between parts accelerator and parts catalyst to be added to 100 parts of a resin, when the ratio of accelerator to catalyst is given.

time of two hours. This is Step 1. Other graphs are made up for different gel times.

2. For Step 2, the operator locates on Figure 1 the accelerator concentration he has been using, and lays a ruler within this marked area so that it connects the observed relative humidity at the top of the area with a proportionally spaced mark across the bottom of the particular accelerator area.

3. Step 3 consists of moving horizontally from the observed temperature at the left until this line meets the ruler.

4. From this point the operator, for Step 4, moves vertically down to the bottom line to read off the recommended accelerator/catalyst ratio.

5. For Step 5, the operator calculates the per cent catalyst to use, or consults a chart such as Figure 2, where he finds a slanting line corresponding to the per cent accelerator. From the point where this line intersects a vertical line from the recommended accelerator/catalyst ratio, the operator moves to the left to find the corresponding catalyst percentage to use to obtain the desired gel time at the observed temperature and relative humidity.

Figure 2 can be used for any catalyst and accelerator since it shows only an arithmetical relationship between parts accelerator and parts catalyst to be added to 100 parts of a resin when the ratio of accelerator to catalyst is given.

6. Step 6 would cover the option of a change in gel time desired for one reason or another. A much longer or shorter gel time might involve not only a change in per cent catalyst but also a change in per cent accelerator. This is because a speeding of the gel time can be achieved by an increase in either accelerator or catalyst. Some resin suppliers, however, recommend ranges of accelerator/catalyst ratios because, outside of these ranges, optimum physical properties are not easily achieved. Therefore, for a change in gel time, a chart such as Figure 1 is picked for the new gel time and an accelerator/catalyst ratio in the supplier's recommended range is chosen which will intersect the observed temperature line within the area of one of the graphed accelerator percentages. The relative humidity adjustment is made again to give a slightly changed accelerator/catalyst ratio, and this is plugged into Figure 2 as before to find the percentage catalyst.

The particular chart chosen for Figure 1 was obtained from data on a definite trio of resin, accelerator, and catalyst. Obviously such a graph would have to be available in the technical or manufacturer's literature, or be determined by the operator for the resins, humidities, and other elements with which he wished to work. Since such data are not available, it is important that the rather simple method of obtaining them be recorded here. Even the data obtained during the present study cover only a few resins and only one resin was employed in the complete range of the variables tested.

RECORD SHEET FOR OBTAINING DATA TO CONSTRUCT WORKING GRAPHS

RUN NO.: G-3-5 VARIABLE: catalyst CONSTANT: accelerator
DATE: 2/13/59 Drops constant: 73.0/g Wt. %: 2.0%
CATALYST: Lupersol DDM Gms/Drop: 0.0137
ACCELERATOR: 6% Co Naphth. Gms/Drop: 0.1230
RESIN: Celanese MR-28C Viscosity: 6.0 poises @ 72°F Wt.: 50.0g

Sample No.	Wt. Resin Present	Drops of Variable Added	Total Gms. Variable Present	Wt. % Variable	Time Variable Added	Time Sample Gelled	Gel Time, Mins.	Ratio Accel. to Catalyst
1	50.0	10	0.1370	0.274	10:21	too long		7.30
2	49.9	3	0.1785	0.357	10:23	too long		5.61
3	49.8	7	0.275	0.552	10:24	12:35	131	3.63
4	49.7	7	0.370	0.743	10:25	11:32	67	2.69
5	49.6	6	0.453	0.914	10:26	11:11	46	2.19
6	49.5	14	0.644	1.30	10:27	10:50	23	1.54
7	49.4	20	0.919	1.86	10:28	10:42	14	1.08
8	49.3	33	1.370	2.78	10:29	10:37	7.5	0.72

Weight container plus remaining resin	51.0g
Weight container	1.0g
Weight resin remaining at end of run	49.1g
Weight resin removed	0.9g
Weight resin removed per sample 0.9/8	0.1g
Average dry bulb temperature:	72°F
Average wet bulb temperature:	67°F
Average relative humidity:	78%
Av. grains water/lb dry air:	91gr/#

Figure 1 is the graph for a two-hour gel time under conditions of high humidity where most of the troubles are encountered. This, and additional graphs covering wide ranges of the variables, were made using data from the MR28C resin of the Celanese Corporation of America, 6 per cent cobalt naphthenate solution accelerator of the Nuodex Products Co., and Lupersol DDM catalyst from Wallace and Tiernan, Inc. Percentage values on these graphs are parts by weight of these solutions added to the weight of resin.

The Procedure

A typical record sheet which can be followed while examining the procedure used to obtain data for these graphs is shown. When, as in the present development of these graphs, no suitable graph is available for modification, the procedure is as follows:

1. In a room of known and fairly constant humidity and temperature, preferably completely air-conditioned and adjustable, all materials are stored for several days. Densities of the catalyst and accelerator, if not available from the manufacturer, are determined by hydrometer or pycnometer. Several 1.0-ml hypodermic syringes are calibrated, and the weight of a drop of each solution is determined.
2. Using a 50.0-g sample of resin in each of eight paper cups, drops of accelerator from the syringe are stirred into each to give a range of 0.3 per cent to 3.0 per cent solutions.
3. The desired number of drops of catalyst for the lowest percentage is then stirred rapidly into one of the accelerated resin batches and the time recorded. Depending on viscosities, a consistently sized sample from 0.1 to 0.2-g for gel testing is removed by a small spatula and flowed onto spaces marked on a piece of cellophane spread over a glass plate. Seven more samples of catalyst are added successively to the same accelerator/resin batch, and gel samples removed after each stirring, to cover a range of 0.3 to 3.0 per cent catalyst. With practice, only one minute is required to prepare each gel sample. Additional research tests were made to show that differences in gel time between this method and that where separate batches of resin are used for each combination, were within the experimental error of determining the gel time. The process is repeated for each resin-accelerator batch. Of course, the catalyst could be kept constant and the accelerator varied if desired.
4. With practice, a standard amount of resin solution can be removed for each gel test. A weight check at the end of the run can be used to calculate closely the per cent catalyst in each gel sample withdrawn.
5. After a series of gel samples is deposited on the cellophane, the gel time tests must be started immediately on the most concentrated samples and tests are run rapidly back through the slowest curing sample. The time of gelling is recorded as when the sample, upon being stroked and worked with the spatula, will show some coagulated lumps and viscous strings which begin to break and no longer fall back on the resin button to form a smooth surface. Since at this point the resin could not have been removed from the supply pot nor worked on the laminate structure, this time is recorded as the gel or practical cure point. When the whole series of tests was repeated on the same day at the same temperature and humidity, deviations of less than 10% between corresponding gel times were obtained.
6. The foregoing series of tests should be repeated under enough variations of temperature and relative humidity to cover the range desired and the resulting data graphed as in Figure 1. The present study has indicated that accurate graphs can be obtained from data obtained at 10-degree temperature intervals and 10 per cent relative-humidity intervals.

Additional Observations

The results of these tests gave straight lines on semilogarithmic paper except at very high and very low temperatures outside of any conceivable working conditions. Humidity effects were practically linear on these graphs and can be extrapolated to lower values.

There is one short cut which appears possible in setting up the graphs for a new resin-curing system, and which is recommended for further study. It appears that the data for many polyester resins may give practically the same slopes when graphed. This makes it possible to use the data for only one point on any line, plus the slope, to determine the line for a new resin in comparison with a resin being studied, such as the MR28C investigated here. In any case, all polyesters studied so far give straight lines so, it is believed, a maximum of two points of data will determine a particular line when graphed as in Figure 1.

Several experiments were run to determine the effect of sample size on gel time. It is well known that exothermic reactions are speeded when heat is not removed as rapidly as it is generated. The larger the volume/surface ratio of the reaction mass, the shorter the gel time should be. Therefore, unless a practical sized batch of catalyzed resin is kept close to room temperature by placing it in a shallow pan or by cooling it, the experimental gel time on the small test sample will have no practical significance. For instance, one 50-g batch cured twice as fast as the sample on the cellophane. The effect of humidity on the sample corresponding in thickness to that which would be used in making the laminate was substantiated here, also. The above test run (at 93 per cent humidity) was repeated at 54 per cent to give a gel time only 25 per cent faster for the 50-g batch. A large number of tests covering the ranges in temperature, humidity, accelerator, and catalyst substantiated the relative effect of humidity on the gel times of small and large samples. In commercial operations the problem of premature gelling of large batches can be solved by mixing the catalyst in one part of the resin, and the accelerator in the rest of the material. Simultaneous coating of the glass cloth with the proper proportions of these two solutions produces a laminate with a gel time corresponding to that of a small sample of the same thickness. Tests on such solutions run over several months' time showed that either the catalyst or the accelerator will increase the viscosity of the raw resin, but not enough to be of practical significance.

Mathematical Procedures

Commercial polyester resins are mixtures of large molecules prepared by esterification of alcohols containing more than one hydroxyl group with acids containing more than one carboxyl group to split off water. This is followed by a separate cross-linking step of true polymerization of double bonds contained in some of the alcohol and/or acid molecule residues, or in subsequently added unsaturated monomers. The polymerization reaction can be hastened by the use of catalysts and their accelerators to the point that the reaction will take place at room temperature in a reasonable length of time.

The velocity of this polymerization of catalyzed polyesters is influenced by the ambient temperature and humidity, as well as by the other previously mentioned variables which usually are fairly well controlled.

The literature reveals that some aspects of these reactions have been studied to correlate certain data. For instance, commercial bulletins such as those from the Pittsburgh Plate Glass Company on Selectron 5000 give the gel time by a peak exothermic temperature test according to the method of the Specifications of Standards Committee, Reinforced Plastics Division, Society of the Plastics Industry. The Interchemical Corporation in its bulletins shows the exotherm curve for each resin and gives the amount of certain

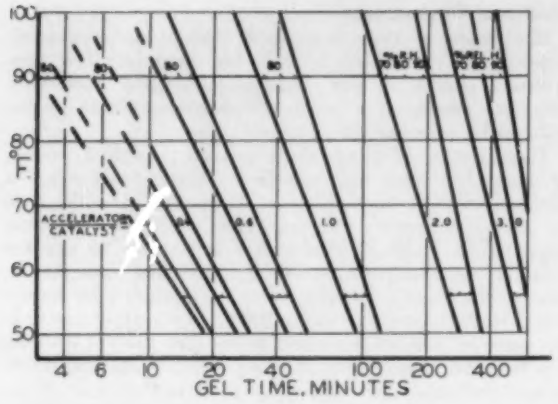

Figure 3. Parameters of the related humidity and of the accelerator/catalyst ratio.

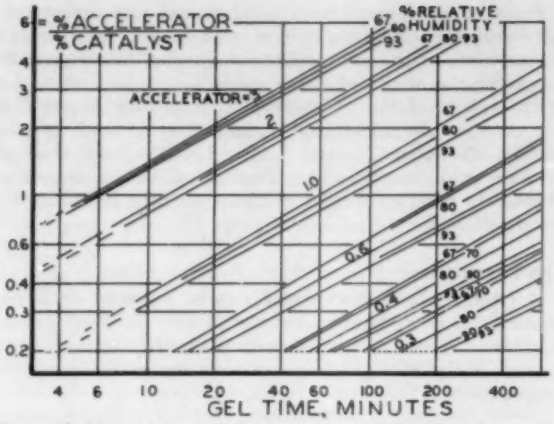

Figure 4. Humidity and percentage accelerator shown as parameters.

catalysts for definite gel times. The American Cyanamid Co., as in the bulletin for Laminac 4116, gives gel times for ranges of catalyst and accelerator. Hooker Electrochemical Co. gives graphs relating catalyst concentration and temperature for various gel times in Bulletin No. 51, while a different test for gel time is mentioned in the Rohm & Haas Co. bulletin on Paraplex P-444, and correlated with some values for temperature, catalyst and accelerator. Several bulletins mention the retarding effect of high humidity but it is believed that data for this effect and correlation of it with the other variables is not available in the industrial literature. Many pertinent references of a general nature are available (1, 2, 7, 10, 13, 14, 15), some of a theoretical nature (5, 6, 9, 11), while some mathematical correlations have been reported (3, 4, 8). A complete study, however, of the correlation of sufficient variables to control the cure or gel time has not been found, nor have empirical equations been derived except those relating a few variables as reported by Berndtsson and Turunen (3, 4), and Maltha and Damen (8). The former investigators found that there were upper limits of effectiveness for either catalyst or accelerator, and derived an equation which correlated the rate of polymerization with the concentration of catalyst. The effects of accelerator and temperature were not included in the equation, nor was the effect of humidity investigated. Maltha and Damen (8) conducted a similar study at a lower temperature to show that a different constant was involved.

Developing The Data

A series of straight lines was obtained when the ratios of percentages of accelerator to catalyst were plotted versus gel time in minutes at each of several constant temperatures. Parameters of percentage accelerator and of relative humidity indicated that humidity values could be interpolated and to a conservative extent extrapolated. By cross plotting, using temperature on the arithmetic scale versus gel time on semilogarithmic paper, a series of lines was obtained for each percentage of accelerator as shown in Figure 3. Parameters of the related humidity and of the accelerator/catalyst ratio are shown. Figure 3 is for an accelerator percentage of one per cent.

Replotting the smoothed data at 10°F intervals resulted in the series of lines shown in Figure 4 where accelerator/catalyst ratio is the vertical coordinate versus gel time in minutes with both humidity and percentage accelerator as parameters. Figure 4 shows the results at 70°F.

Another straight-line relationship was obtained when relative humidity at a constant temperature and a constant ratio of accelerator to catalyst was plotted against gel time, while the percentage of accelerator was used as a parameter. Figure 5 shows that this would be a convenient type of chart to predict gel time of a standard resin mixture at different relative humidities.

When a constant gel time is required, a chart like that shown in Figure 6 is convenient. Relative humidities and percentage accelerator serve as parameters when temperature is plotted versus accelerator/catalyst ratio. This type of chart and an accompanying mathematical conversion graph showing the relation between percentage catalyst and percentage accelerator for varying accelerator/catalyst ratios were used in the first part of this article.

Mathematical Correlation

In the initial plots of the data (as in Figure 4 at 10°F. intervals on logarithmic paper with the ratios of percentage of accelerator (A) to catalyst (C) versus gel times (theta), the resulting straight lines could be described by the equation:

$$A/C = a(\theta)^{b} \qquad \text{(Equation 1)}$$

where the constant (a) would represent the intercept of the line on the ordinate when

$$\theta = 1 \text{ (and } \ln\theta = O)$$

and the (b) constant would be slope of the line. The values of the a and b constants were determined for lines covering a range of the parameters of relative humidity and percentage accelerator. The following calculations illustrate the method used to determine ranges of values for these two constants:

$$\text{If } A/C = a(\theta)^{b}, \text{ then } \ln(A/C) = b \ln \theta + \ln a$$

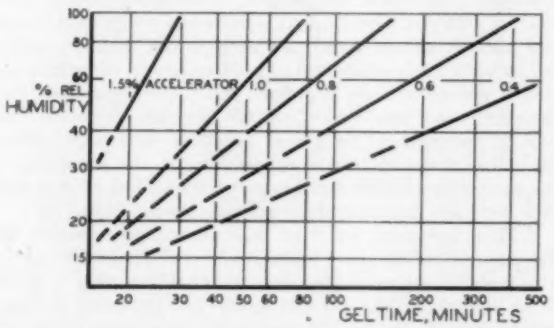

Figure 5. A convenient type of chart to predict gel time of a standard resin mixture at different relative humidities.

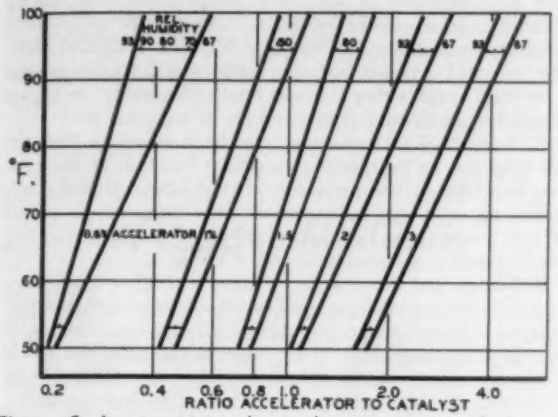

Figure 6. A convenient chart when constant gel time is required.

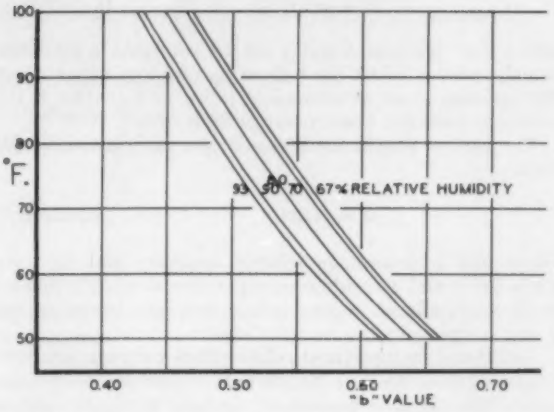

Figure 8. Temperature vs. "b" constant.

From the original data, (which is too lengthy to present), when the accelerator value is one at 70°F. with a relative humidity of 67 percent and an A/C ratio of one is chosen, the gel time θ_1 is found to be 57.5 minutes. Similarly for an A/C ratio of four, the gel time θ_4 is found to be 625 minutes. Substituting these values in the logarithmic equation the two resulting equations can be solved by eliminating ln a and solving for b.

$$\ln (A/C)_4 = b \ln\theta_4 + \ln a$$

$$\ln (A/C)_1 = b \ln\theta_1 + \ln a$$

$$\text{so, } \ln (A/C)_4 - \ln (A/C)_1 = b \ln\theta_4 - b \ln\theta_1$$

$$\text{or, } \ln (4/1) = b \ln(625/57.5), \text{ and } b = (\ln 4)/(\ln 10.87)$$

$$= 1.386/2.385 = 0.580$$

In order to find the values of the a constants, the logarithmic version of the equation can be rearranged as $\ln (1/a) = b \ln\theta - \ln (A/C)$. When $A/C = 1$, $\theta = 57.5$, then $\ln (1/a) = (0.580) \ln 57.5$, and $a = 0.0953$.
Therefore for accelerator percentage of one, temperature of 70°F. and relative humidity of 67 per cent:

$$A/C = 0.0953(\theta)^{0.580}$$

Similarly the constants for other combinations of variables were calculated and a family of graphs (Figure 7) was constructed to show the relationship between the variables by plotting temperature versus the "a" constant with parameters of relative humidity and percent accelerator with the constant b representing the slopes of the lines. It was found that this slope was practically constant for a given temperature and relative humidity for all values of per cent accelerator. Therefore, it was possible to construct another series of lines, shown in Figure 8, by plotting temperature versus the constant b with relative humidity as the parameter. Thus, the a and b constants can be determined for any set of values of temperature, humidity, and per cent accelerator. The per cent catalyst is then obtained from the equation

$$A/C = a(\theta)^b.$$

This equation can be related to the Berndtsson and Turunen (3) equation: $\theta = K(C)^n$ (which can be converted to the form $1/C = a(\theta)^b$) by realizing that $K^{-n} = a$, $n = b$, and $1/C = \theta^{n}K^{-n}$ when the accelerator concentration is kept constant.

In the same manner the graphs in Figure 3 could be used to determine constants for the equation of these lines which would be of the form:

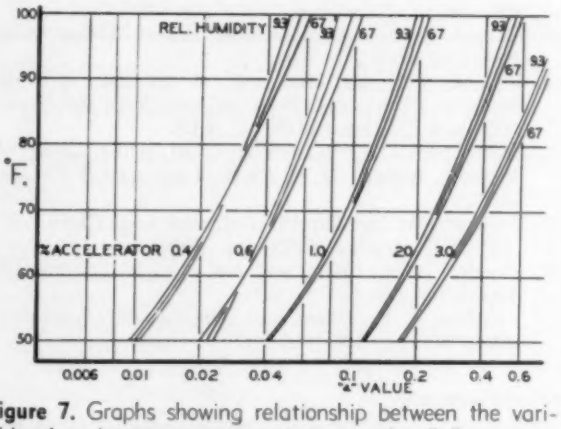

Figure 7. Graphs showing relationship between the variables by plotting temperature versus the "a" constant with parameters of relative humidity and percent accelerator with the constant "b" representing the slopes of the lines.

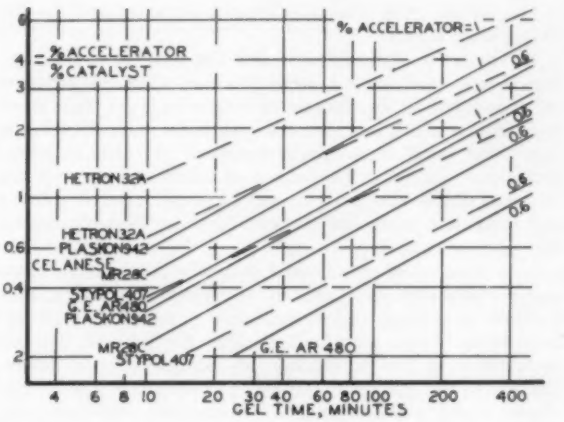
% ACCELERATOR = % ACCELERATOR / % CATALYST
HETRON 32A
HETRON 32A PLASKON 942
CELANESE MR28C
STYPOL 407 G.E. AR480 PLASKON 942
MR28C STYPOL 407
G.E. AR 480
GEL TIME, MINUTES
4 6 8 10 20 30 40 60 80 100 200 400

Figure 9. Data for various commercial resins compared with Celanese MR28C resin.

$$\theta = c(e)^{dT} \qquad \text{(Equation 2)}$$

where T is° Rankine, c and d are the constants to be determined, and e is 2.718, the base of natural logarithms. Since this equation is not as convenient to use as Equation 1, the constants have not been investigated further.

The data or graphs for Figure 5 give an equation of the form:

$$\theta = p(RH)^q \qquad \text{(Equation 3)}$$

where RH is percentage relative humidity and the constants are p and q. Additional experimental work is needed in the extrapolated regions before accurate values of the p and q constants can be calculated.

Additional mathematical relationships between the variables are possible. For instance, in Figure 6 an equation and constants can be worked out similarly to give the ratio of the accelerator to catalyst needed to produce a 30-minute gel time at a chosen temperature and humidity. In other works $A/C = r(e)^{sT}$ where T is ° Rankine and r and s are constants.

Resin Variable

A few of the data on other commercial resins are compared in Figure 9 with the corresponding data for Celanese MR28C which was used in the majority of the experiments. It can be seen that those resins giving a similar slope will require only a change in the a constant to correlate their data with those of MR28C, while those with a slope different from that of MR28C can be correlated by determining the values of both a and b constants. The fact that these latter resins (Hetron 32-A of the Hooker Electrochemical Co., Niagara Falls, and Stypol 407 of the H. H. Robertson Co., Ambridge, Pa.) have practically identical slopes and are offered as being more heat resistant than the other resins suggests possible correlations based on chemical structure. The data used in Figure 9 are for 80°F. and 67 per cent relative humidity and only 1.0 per cent and 0.6 per cent accelerator concentrations are shown. Similar correlations were found at other temperatures and at other relative humidities and accelerator concentrations, but in the latter case higher humidities at lower accelerator concentrations tended to give lines with lower slopes which would necessitate a change in the b constant for the most accurate work. Also observed was the fact that a change in humidity had more effect on some resins than on others.

Conclusions

The results from this experimental work suggest the following conclusions:

1. The work of Berndtsson and Turunen (3, 4) was confirmed in that there is a maximum useful concentration of either catalyst or accelerator beyond which gel time is not appreciably shortened. In the particular case studied in detail this point is at approximately 2 per cent of added accelerator solution. The effect of additional catalyst fell off more slowly above 1 per cent of added catalyst solution and then only when the percentage of accelerator solution was greater than 1 per cent.

2. A number of commercial polyester resins were tested under controlled variations in the relative humidity, temperature of gelling, and percentages of added catalyst and accelerator. These tests indicate that the ratio of the concentrations of the accelerator to catalyst bear a definite relation to the gel times through the equation $A/C = a(\theta)^b$ where a and b are constants characteristic for each resin. This has been shown to be an expansion of the Berndtsson and Turumen equation $\theta = K(C)^n$ where K and n are constants, to include the effect of the accelerator. The constant b for each resin was found to be the same for all accelerator concentrations at any fixed temperature and relative humidity. It increased as either temperature or relative humidity was decreased, varying between 0.42 and 0.66. The constant a varied between 0.006 and 0.70. In general it increased with either lowered relative humidity, or higher values for accelerator concentration or temperature.

3. Additional relationships have been found in that the gel time can be expressed as a power function of the relative humidity in the form $\theta = p(RH)^q$ where p and q are constants, or it can be expressed as a temperature power of the transcendental number "e" in the form $\theta = c(e)^{dT}$, where c and d are constants and T is °R.

4. Longer gel times were obtained at higher humidities. This may be caused by a reaction of moisture with the free radicals believed to be responsible for the cross-linking of the resin. Increased time of exposure of the resin film would allow more moisture to diffuse into the resin. This is substantiated in part by the greater effect of an increase in humidity at higher temperatures which would make available a higher concentration of moisture in the air. The relatively smaller effect of increased humidity at low temperature even at long gel times could result from the diffusion equilibrium being reached more rapidly at these lower moisture concentrations.

5. The theoretical relationships between the chemical structures and the mathematical correlations of the variables tested remain to be explored.

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Acknowledgment

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Monsanto Chemical Co.

For Epoxies . . .

Improves compressive strength and dimensional stability
Better electrical properties
Adhesives are stronger
Accelerated cure and lower viscosity

A Unique Modifier

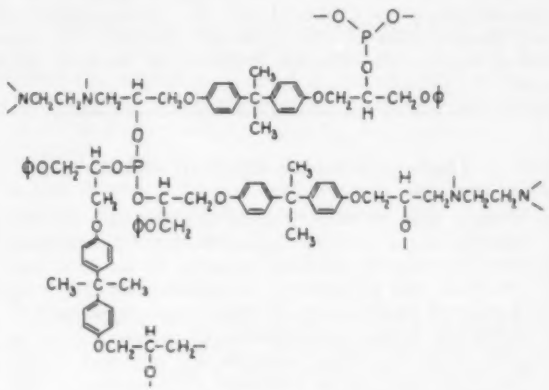
Because of the high cost and viscous nature of liquid epoxy resins, it is desirable for most applications to modify them by the addition of diluents, fillers, and other materials. One such modifier–triphenyl phosphite–has been uncovered, which may open up new facets of resin technology.

Mod-Epox, Monsanto's trademark for triphenyl phosphite for epoxy resin applications, is completely miscible with the normal liquid epoxy resins and can be blended over a range of concentrations to give economy and excellent physical properties. In one study, concentration at 25 parts per 100 parts of resin was found to give optimum properties. Resins containing this modifier may be stored in closed containers for several months without any appreciable change in viscosity. Caution: Triphenyl phosphite is hygroscopic; contamination with moisture will affect the performance characteristics. Formulations open to the atmosphere will exhibit skin formation after several weeks, although resin viscosity under the skin will change at only a very slow rate.

How it works

Triphenyl phosphite enters the polymer molecule through a transesterification reaction. This reaction occurs between the phosphite ester and the resin's hydroxyl groups. Under these conditions, crosslinking would be effected not only through the amine curing agent, but also through the trifunctional phosphite ester, giving an intermediate polymer molecule of the type shown. The triply connected phosphorus atoms of the intermediate molecule then undergo thermal isomerization to quadruply connected phosphorus atoms, i.e., phosphonates. Experiments with other curing agents, such as phthalic anhydride and the polyamides, also substantiated that the modifier becomes chemically bound into the polymer.

Evidence to support the proposed mode of action came from a comparison of the physical properties of cured resins containing varying amounts of modifier and curing agent. When the modifier was added to a resin formulation containing a quantity of polyamine curing agent equivalent to the resin's oxirane oxygen content, some decrease in physical properties of the cured resin occurred. But when the curing agent concentration was cut so that the sum of



Triphenyl phosphite chemically bound in intermediate polymer molecule

TABLE I
Comparative Properties of Formulations

Material	Control	I	II	III	IV
Liquid epoxy resin (E.V. = 200)	100	100 (0.5 mole oxirane)	100	100	100
Triphenyl Phosphite (Mod-Epox)		25 (0.24 mole—0)	25		
Allyl glycidyl ether				10	
Butyl glycidyl ether					13.5
Diethylene triamine	12 (0.58 mole N-H)	8 (0.31 mole N-H)	11 (0.53 mole N-H)	13	13
Cost, ¢/lb. of modified resin*	—	61¢	61¢	71¢	67¢
Formulation Properties**					
Viscosity, cps at 25°C.	1650	920	620	450	420
Hardness, Rockwell M	102	103	93	90	90
Heat Distortion Point, °C.	114	74	78	83	76
Compressive Yield Strength, psi	15,550	16,500	***	14,100	13,250
Water Absorption, % in 7 days	0.40	0.38	0.58	0.50	0.52
10% Caustic absorption, % in 7 days	0.31	0.32	0.54	0.40	0.42

* Based on prices published Sept., 1959.
** Cure schedule: 20 hours at room temperature plus 2 hours at 100° C.
*** No yield point. Slope changed at about 16,000 psi.

the modifier's phenoxyl content and the curing agent's amino hydrogen content about equaled the resin's oxirane content, the physical properties of the cured resin were the same or better, with the exception of the heat distortion point, than those of the control.

The heat distortion point (see Table I) was reduced to a value of approximating as that observed when several of the commercially available glycidyl ether reactive diluents were used at somewhat lower concentrations. This similarity is in line with the mechanism advanced since the phenoxyl groups and glycidyl ethers both act as chain-terminating agents and tend to reduce the degree of crosslinking. Thus, the stoichiometry of resin formulation No. 1 suggests that triphenyl phosphite acts as a substitute for the polyamine curing agent—a unique feature. Also implied: The driving force for the proposed transesterification reaction is capture of the phenol released during the ester interchange step by the resin's excess oxirane rings. The small amount of free phenol present during the reaction also can act as an accelerator in modified systems cured with nitrogen-containing curing agents. This cure acceleration has been observed in some triphenyl phosphite modified resin systems.

Electrical properties of the formulated resins are among the properties which can be improved through the use of triphenyl phosphite (Table II). Of the commercially available reactive diluents only triphenyl phosphite will maintain or improve the electrical properties of the basic epoxy resin.

Triphenyl Phosphite Effect on Adhesives

Tensile tests on steel bonded to steel with an unmodified liquid epoxy cured with 12 PHR diethylene triamine showed the bond strength to be 2000 psi. Modified with 25 parts triphenyl phosphite and using 8 parts of curing agent, the bond strength jumped to 3,060 psi. In tests on aluminum to aluminum bond, a 40% increase in bond strength was observed. Using butyl glycidyl ether as a diluent, a 7% increase in the aluminum-aluminum bond resulted.

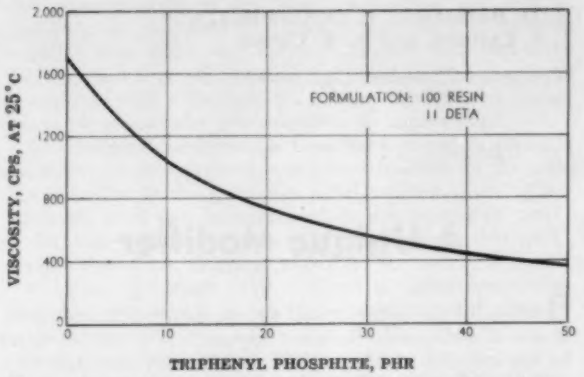


Figure 1. Viscosity curve of an epoxy resin formulation with various amounts of triphenyl phosphite. At the level of 25 parts of triphenyl phosphite, this formulation has a viscosity of about 600 centipoise and shows excellent handling characteristics.

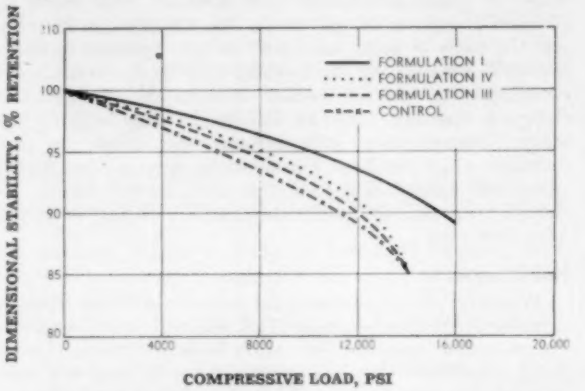


Figure 2. Deformation of castings versus compressive load. At loads of 14,000 psi and higher, the triphenyl phosphite formulation showed 50% better retention of dimension than any of the other resin systems including the unmodified resin. The importance of this feature in formulations designed for the tool and die industry is readily apparent.

TABLE II
Electrical Properties of Various Formulations

Sample Composition	Power Factor (%)		Dielectric Constant		Specific Resistivity*
	1 kilocycle	1 megacycle	1 kilocycle	1 megacycle	Megohm-cm. at 50° C.
100 Liquid Epoxy Resin** 12 Diethylene triamine	1.4	3.6	4.9	4.3	16.6×10^{6}
100 Liquid Epoxy Resin 8 Diethylene Triamine 25 Mod-Epox	0.4	1.7	4.0	3.9	18.8×10^{6}
100 Liquid Epoxy Resin 13 Diethylene Triamine 10 Allyl Glycidyl Ether	1.3	3.5	4.9	4.3	7.1×10^{6}
100 Liquid Epoxy Resin 13 Diethylene Triamine 13.5 Butyl Glycidyl Ether	1.0	3.1	4.7	4.2	7.3×10^{6}

* Specific resistivity measurements at room temperature correlated fairly well with the 50° C. values, but were not included in the table because the observed resistances at room temperature approached the leakage resistance of the bridge too closely to give accurate results.

**Typical resins are Ciba's Araldite 6010. Devoe-Raynolds' Epi-Rez and Shell's EPON 828. Similar liquid resins are also manufactured by Union Carbide Plastics and Reichhold.

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Refractive Index Method for Determining Cure Rates of Epoxy Resins

This article outlines a new method for testing cure performance of epoxy resins and curing agents. Results are presented in the form of rate curves. Effect of curing agent concentration, influence of aqueous, aliphatic, and aromatic hydroxyl groups on the cure rate and the temperature dependence of the reaction are also reported

THE development of the field of epoxy resins has been so rapid that it has been difficult to provide an adequate variety of reliable methods for the evaluation of these novel products. A particularly challenging problem was the design of methods for measuring the rate and extent of the reaction that is commonly called the cure of epoxy resins.

These difficulties, of course, stem from the fact that the resins in question, after addition of curing agent, pass from the liquid state through a gel and rubbery stage to the solid state and eventually become completely insoluble and infusible. The desired methods should be applicable to all these stages, preferably on the same specimen. Another obstacle in carrying out measurements on epoxy resins during the cure is the evolution of heat of reaction, sometimes called "exotherm". Reaction studies should be carried out at constant temperature; therefore, the heat of reaction has to be removed.

Fortunately there are a number of ways to handle this problem. One of the methods (4) circumvents the solidification problem by using a model compound, namely an epoxy compound with a structure similar to the materials to be tested, but not being polyfunctional. Thus, a cure reaction can be carried out and studied by chemical analysis without the occurrence of gelation or solidification. This method has given valuable insight into the reaction mechanism of epoxy polymerizations, but the results are not fully applicable to the reaction of diepoxides such as EPON® resins.

A method of chemical analysis applicable to epoxy resins in the solid state is available (2, 1) but is not practical for specimens in the gelatinous or rubbery state. Infrared spectroscopy has been used for cure rate determinations (2); by sacrificing a pair of rocksalt plates, it is possible to study the cure from inception to completion on the same specimen. Viscosimetric methods and gel time determination are frequently used for cure rate measurement, but they deal with the first part of the cure only and, as will be shown later, are not clearly related to the rate of cure.

This paper deals with the measurement of cure rates by refractometry. When a resin cures, it shrinks, thereby increasing both its density and its refractive index. The increase of refractive index can serve as a measure for the extent of the cure.

The Equipment

An Abbe-type refractometer was used throughout in this work. This instrument combines high accuracy ($\pm$ 0.0001) with ease of handling and thermostating. Furthermore, the sample is protected from access of air and moisture and, most important, such a small amount of sample is used that the exotherm is entirely negligible.

Special provisions were needed to prevent adhesion of the cured resin to the refractometer prisms. Though mold-release agents were usable, their application was difficult and not fully reliable. A better way was found in covering the prisms with pieces of thin plastic film. Polyvinylidene chloride film (Saran) was used over the measuring prism and polytetrafluoroethylene film (Teflon) over the illuminating prism. This arrangement is usable even at high refractive indices, the upper limit being set by the refractive index of the Saran film ($n^{\infty}_{D} = 1.60$). Experimental details of the technique are given in the appendix.

To carry out a cure rate determination the curing agent is added to the epoxy compound and mixed in carefully. Two drops of the mixture are placed on the protected refractometer prism. Measurements are started after one minute and repeated as desired without removing the specimen.

If determination of the gel time is desired, this is done on a few drops of the mixture in a test tube immersed in the thermostat that serves the refractometer. This method is described in the appendix.

Presentation of Results

Piperidine was the preferred curing agent in the initial part of this work because of its conveniently slow reaction rate. Some representative data obtained with this and other curing agents appear in Table 1. It should be noted that there is, in most cases, an immediate increase of the refractive index over the value calculated from the refractive indices of the components. This is probably caused by hydrogen bonding of the amine and ceases after a few minutes. In the table, values for "Refractive Index at Mixing Time" and "Refractive Index at Zero Time" are given, the former referring to values calculated from the components, the latter to values obtained from experimental data by extrapolation in such a way that the hydrogen bonding effect is eliminated. Values for the increase of refractive index from "zero time" to "gel time" and from "zero time" to "final resin" (i.e., after 24 hours at 65°C) are also given in the table.

Two facts of interest are apparent: First, the total increase of the refractive index (from "zero" to "final") is nearly the same for all secondary and tertiary amines, namely approximately 280 units of the 4th decimal place, whereas primary amines give higher values of approximately 400 and a mixed primary-tertiary amine an intermediate figure of 360. This is explained by different reaction mechanisms; primary amines form C-N bonds, but secondary and tertiary amines predominantly form C-O bonds in the process of curing. And secondly, the gain from "zero time" to "gel time" varies considerably for different curing agents and does not correlate well with the total gain. It was concluded that gel times should not be used to describe cure rates.

Table I. RATE OF CURE OF EPON 828 WITH AMINE CURING AGENTS

Curing Agent		Refractive Index, n_D^{25}				Gain of R. I. n_D^{25}				
phr[a]	Substance	At Mixing Time	At Zero Time	At Gel Point	Final After 24 hrs.	Mixing To Zero	Zero To Gel	Zero To Final	Half Time Of Cure[c]	Gel Time[c]
2	Piperidine, Tech.	1.5526	1.5540	1.5574	1.5800[d]	14	34	260	12:05	5:33
4	Piperidine, Tech.	1.5503	1.5531	1.5579	1.5810	28	66	279	5:40	3:50
6	Piperidine, Tech.	1.5480	1.5523	1.5600	1.5800	43	77	277	4:15	3:05
8	Piperidine, Tech.	1.5458	1.5517	1.5597	1.5790	59	80	273	3:10	2:30
5	Piperidine, Pure	1.5491	1.5540	e)	1.5815	49	e)	275	5:00	e)
5	Piperidine, Pure	1.5491	1.5539	1.5643	1.5815	48	104	276	4:55	4:10
+1	Water									
5	Piperidine, Pure	1.5469	1.5527	1.5574	1.5814	58	47	287	3:35	2:20
+1	Water									
5	Piperidine, Pure	1.5469	1.5521	1.5575	1.5811	52	54	290	3:20	2:15
+5	Phenol									
5	Piperidine, Pure	1.5483	1.5536	1.5668	1.5835	53	132	299	2:14	2:04
+5	Bisphenol—A	—	1.5546	1.5648	1.5816	—	102	270	2:25	2:00
5	Piperidine, Pure, Dry[f]	1.5471	1.5521	1.5620	1.5800	50	99	279	15:10	10:20
4	Triethylamine	1.5483	1.5493	e)	1.5800	10	e)	307	3:01	e)
4	DMP—30[j]	1.5529	1.5588	e)	1.5860	59	e)	272	1:23	e)
8	Diethylaminopropylamine[f]	1.5428	1.5398	1.5530	1.5758	—30	132	360	1:20	1:01
7.1	Diethylaminopropylamine[f]									
+0.9	Water	1.5429	1.5425	1.5563	1.5789	— 4	138	364	1:00	0:43
5	Ethylene diamine	1.5494	1.5499	1.5702	1.5860[g]	5	203	361	0:20	0:25
10.4	Hexamethylenediamine	1.5441	1.5368[h]	1.5628	1.5796	—73	255[h]	428[h]	0:24	0:28
12.4	m-Phenylenediamine	1.5634	1.5645	1.5840	1.6052	11	195	407	2:20	2:20
12.6	m-Phenylenediamine	1.5635	1.5653	1.5861	1.6055	18	208	402	2:24	2:27

a) Parts per 100 parts of EPON (by weight).
c) In hours and minutes.
d) After 48 hours.
e) Not determined.
f) In these experiments, distilled EPON 828 was used.
g) After 77 hours.
h) Estimated value.
i) $\Delta n_D^{25} \times 10^4$.
j) Tris(dimethylaminomethyl)phenol.

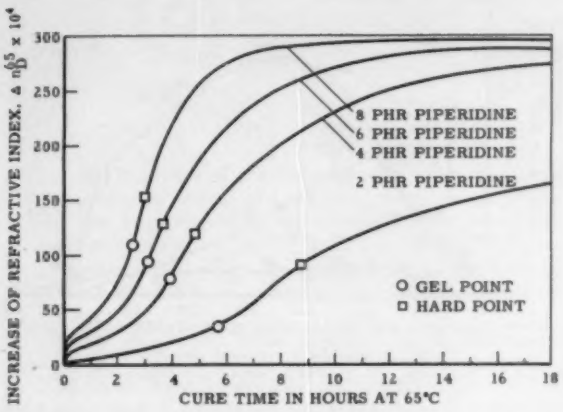
INCREASE OF REFRACTIVE INDEX, Δ n_D^{25} x 10^4
8 PHR PIPERIDINE
6 PHR PIPERIDINE
4 PHR PIPERIDINE
2 PHR PIPERIDINE
O GEL POINT
□ HARD POINT
CURE TIME IN HOURS AT 65°C

Figure 1. Cure of Epon 828 with different amounts of curing agent.

Refractive Index Curves and Cure Rate Curves

Figure 1 shows curves obtained by plotting the increase of refractive index of various mixtures of Epon 828 and piperidine versus time. S-shaped curves resulted. Presentation of curves not conforming to a constant slope are difficult to compare.

A more meaningful way of expressing the result of a cure experiment consists in reading from the refractive index curve (such as shown in Figure 1) the time required to increase the refractive index to a value halfway between zero time value and final value. This time is called "half time" and is shown, together with the gel time, in the last columns of Table 1. The half time expresses something related to the cure rate by a finite number, regardless of the shape of a particular cure curve.

A better way of evaluation is possible by plotting the "cure rate" expressed as the increase of refractive index per minute against the cumulative gain of refractive index, also called "conversion". Curves of this type are shown in Figures 2, 3, 4, 6, 8, and 9. Note that in this type of presentation the reaction time does not appear as a coordinate; however, the reaction times are indicated by markers and numerals along the curve. Gel points are likewise indicated on these curves.

Primary Amines. Figure 2 shows the rate curves obtained with Epon 828 and 2 diprimary amines, ethylenediamine and hexamethylenediamine. Commencing at a very rapid rate, they lose speed rather quickly, even before the gel point is reached, and thence degenerate to a very low rate which continues for a long period of time.

A primary aromatic amine, m-phenylenediamine, on the other hand, performs quite differently; its cure rate curve, Figure 4, is similar to that of a secondary or tertiary amine.

Secondary Amines. Cure rates of Epon 828 with 2, 4, 6 and 8 phr (parts per hundred parts of resin) of technical piperidine are presented in Figure 3. Points obtained after equal cure times have been connected by broken lines. These curves indicate clearly that the curing agent piperidine has an autocatalytic effect; the rate increases during the first hours, well beyond the gel point, goes through a maximum and decreases after solidification has taken place. Finally, though after very different times, all curves arrive at the same point indicating that the final extent of cure is independent of the amount of curing agent.

Tertiary Amines. Tertiary amines have cure rates similar to secondary ones as illustrated by the rate curve for triethylamine, Figure 4.

Influence of the Amount of Curing Agent

An example for the effect of the amount of curing agent has been given in Figures 1 and 3. If one determines the half time for these curves and plots the reciprocal half time against concentration of the curing agent, a straight line is derived. (Figure 5.) Graphs of this kind can be used to determine the most desirable concentration of a curing agent.

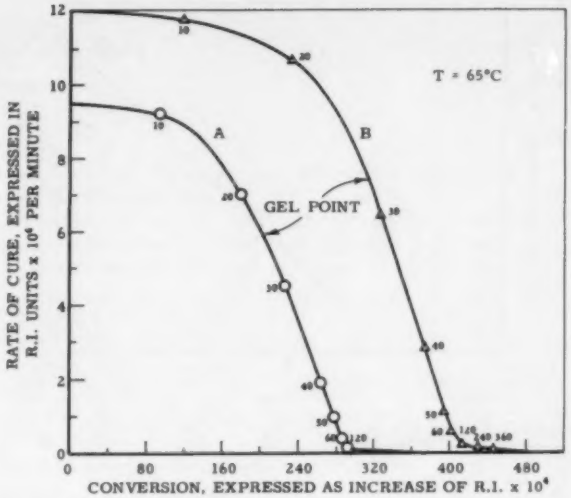

Figure 2. Cure rates of primary amines.
A. Epon 828 + 5 PHR ethylenediamine.
B. Epon 828 + 10.4 PHR hexamethylenediamine.

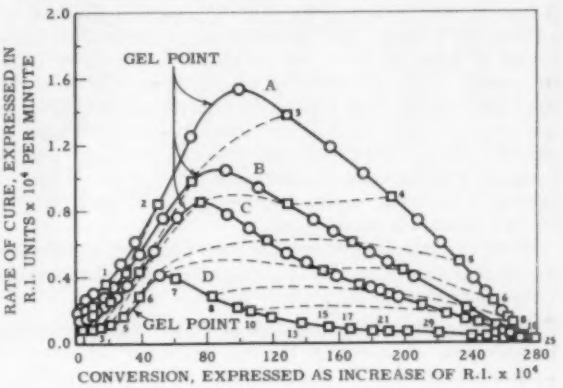

Figure 3. Cure rates of compositions with different amounts of curing agent.
Epon 828 and technical piperidine (A) 8 PHR; (B) 6 PHR; (C) 4 PHR; (D) 2 PHR. Numbers on curves indicate cure time in hours (squares: full hours; circles: fractional hours). Dashed lines connect points of equal cure time.

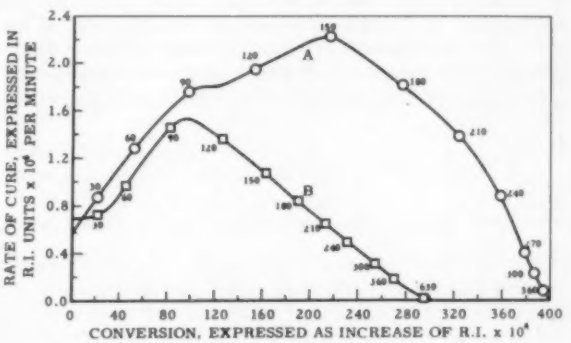

Figure 4. Cure rates at 65°C.
A. Epon 828 + 12.6 PHR m-phenylenediamine. B. Epon 828 + 4 PHR triethylamine. Numbers on curves indicate cure time in minutes.

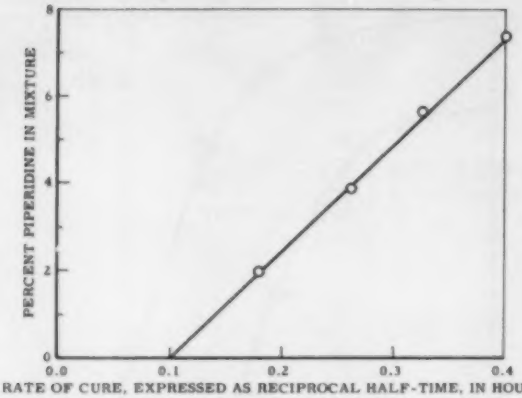

Figure 5. Rate of cure as a function of catalyst concentration in the system Epon 828 + technical piperidine.

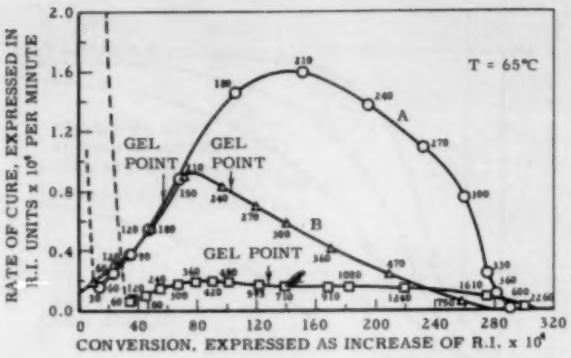

Figure 6. Cure rates with secondary amine and different hydroxyl contents.
A. Epon 828 + 5 PHR piperidine + 1 PHR water. B. Epon 828 + 5 PHR piperidine.
C. Purified Epon 828 + 5 PHR piperidine. Numbers on curves indicate cure time in minutes.

Influence of Hydroxyl Groups. When the curing agent is a secondary or tertiary amine, the influence of hydroxyl groups on the cure rate becomes very prominent (3, 4). Hydroxyl may be present in the form of water, as aliphatic alcohol or as phenol. The refractive index method was employed to study such systems, as shown in Figure 6. Here, the middle curve refers to the system Epon 828 + 5 phr piperidine; addition of 1 phr water increased the cure rate as shown in the upper curve, whereas removal of some of the aliphatic hydroxyl by purification of the epoxy resin diminished it to the values of the lower curve. Again, a plot of reciprocal half time versus hydroxy value (Figure 7) permits a quantitative presentation.

When phenols are present, the cure starts out rapidly, decreases to a minimum, then increases to a maximum and decreases once more as shown in Figure 8.

Acceleration and Deceleration of Cure. The described autocatalytic character of the cure curves is explained by the promoting effect of hydroxyl groups on epoxy polymerization by secondary or tertiary amines. If the hydroxyl concentration is small at the beginning of the cure, a low initial reaction rate is obtained. However, this reaction creates new hydroxyl groups which will catalyze the reaction and thus induce an autocatalytic behavior.

The decrease of the cure rate is a result of two factors, the decreasing concentration of unreacted epoxide, and the immobilization of reactive molecules by solidification of the resin. The second factor prevents the reaction from going to completion or causes an extremely slow end reaction.

Influence of Temperature. The described experiments were performed at 65 ± 0.1°C under conditions which assured the absence of exotherm effects. The system Epon 828 + 5 phr ethylenediamine was also studied at 45.0 and 25.0°C. The results are presented in Figure 9. As before, numerals on the curves refer to cure times, and broken lines connect point of equal cure time. Diagrams of this form permit to work out favorable cure schedules involving successive steps at increasing temperatures.

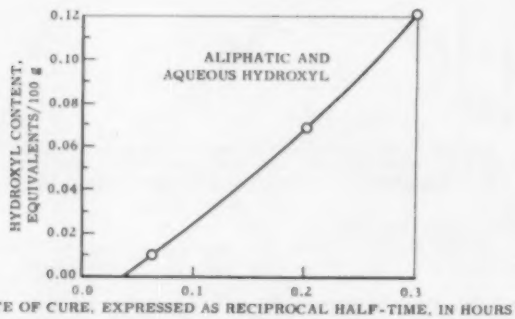
HYDROXYL CONTENT, EQUIVALENTS/100 g
ALIPHATIC AND AQUEOUS HYDROXYL
RATE OF CURE, EXPRESSED AS RECIPROCAL HALF-TIME, IN HOURS⁻¹

Figure 7. Influence of the presence of hydroxyl on the rate of cure of Epon 828 with 5 PHR piperidine.

Relation Between Refractive Index and Chemical Conversion

Up to this point, the assumption has been made that the increase of refractive index parallels the progress of the cure and can be used as a measure for the extent of cure. The question remains to be answered whether there is a precise correlation or even proportionality between the refractive index and the decrease of the epoxy content, the latter being a measure for the degree of cure from the chemical aspect, i.e., disregarding complications by cross-linking. To test this relationship, epoxy determinations were made by infrared spectroscopy during the cure of an epoxy resin and simultaneously refractive index values were obtained on the same sample. The results appear in Figure 10. The two curves obtained are similar, i.e., they are S-shaped, reach a half-way point after 5 to 6 hours and a final value after 30 to 50 hours. However, there is no evidence that the gain of refractive index is proportional to the conversion.

If one considers the allover effect of the cure including cross-linking, one should also compare the gain of refractive index with the softening point of the cured resin. A curve of this type is included in Figure 10; the softening temperatures were obtained on a hotpoint indentation tester (2).

Simplified Method

While the described method will be useful for a number of purposes, especially to study and compare curable materials, curing agents and cure promoters, and to cast a light on the reaction mechanism of such systems, it may be too tedious for other applications, especially those where the performance of a large number of tests is desired.

For such cases, a simplified method has been designed which offers the advantages, over the described method, of not utilizing a refractometer for unduly long times, not requiring excessive manpower to take frequent readings, and not being difficult to perform by an unskilled operator. When this method is used, only two refractive index measurements are taken, the first one after the initial, frequently anomalous, period of the cure has passed and the other be-

The Procedure—Cure Rate

(Complete Method)

Bring prisms of the refractometer to desired temperature by means of circulating warm water. Place a few drops of volatile contacting liquid (petroleum ether at 25°C, isopropyl alcohol at 45 or 65°C) on the measuring prism. Place a piece (2 x 1¾ inches) of ½ mil Saran film (a) on prism and press down with a finger to remove wrinkles and excess liquid. Allow contacting liquid to evaporate for about ½ hour. The film should cling to the prism without showing wrinkles or blisters. Place a rectangular paper frame (1½ x 1¾ in. outside, 1¾ x ½ in. inside, cut from writing paper, .004 in. thick) over the Saran film. This serves as a spacer. Place two drops of the sample to be measured on the Saran film inside the frame. Cover with Teflon film (2 x 1¾ x 0.002 in.) in such a way that the rougher side of the film is toward the sample and the striation of the film parallel to the smaller side of the prism. (b) Close the prisms and start measurements immediately. If the field is too dark, use a strong flashlight held directly against the illuminating prism. As an alternative, indirect illumination can be used as per instructions supplied with the refractometer.

Toward the end of the cure, especially in the presence of curing agents of high refractive index, interference fringes (c) will be observed. They decrease the accuracy of the measurement slightly; the use of a polaroid filter over the ocular of the refractometer, when oriented in the proper direction, was found to improve the situation considerably.

[a] The grade of film used for this work is apt to become electrically charged and, in this condition, picks up dust rapidy. To avoid this, use fresh film from original roll.

[b] The opacity and striation of the film are essential; they increase the aperture of the incident light to the necessary values.

[c] The fringes are probably caused by interference of a light beam coming from the sample/Saran interference with another one coming from the Saran/glass interface. Two sets of fringes appear as the result of the birefringency of the Saran film and appear superimposed. The polaroid filter eliminates one of the sets, according to position.

The Procedure—Gel Time

A few drops of the sample are placed into a test tube of 75 mm length and 8 mm internal diameter. A glass rod of 100 mm length and 7.0 to 7.2 mm diameter with a well rounded end is inserted, thus forming an epoxy resin layer of not more than ½ mm thickness between rod and wall. The tube is inserted in the water bath that controls the temperature of the refractometer and checked at frequent intervals by pulling the rod. Appearance of severe stringiness indicates the gel point. This test is run in duplicate, the second tube being left untouched until the first one shows gelation.

The Procedure—Cure Rate

(Simplified Method)

Weigh 10.00 g epoxy resin into 30 ml beaker. Suspend beaker inside 4 oz jar and fill space around beaker with oil. Place jar in oven until epoxy resin is at 65.0°C.

Weigh out 1.40 g of pure m-phenylenediamine on a piece of Teflon film. Remove jar with beaker from oven, add phenylenediamine, start timer, stir for 4 minutes holding jar in hand in such a manner that the paddle wipes sides and bottom of beaker. The oil jacket will maintain the temperature above 60°C ascertaining ready solubility of the curing agent.

Place two drops of the mixture on the prism of the refractometer. Be sure that the water has been circulating at the correct temperature for at least 30 minutes to bring prisms to temperature.

Take first reading of refractive index (n_D^{65}) exactly 10 minutes after addition of the curing agent. ($n_{10\ min}^{65}$)

Take a second reading exactly 70 minutes after addition of the curing agent. ($n_{70\ min}^{65}$). Clean refractometer prisms immediately with methyl isobutyl ketone.

Report

Refractometric cure rate = $(n_{70\ min}^{65} - n_{10\ min}^{65})\ 10^4$.

fore the sample gels. Obviously, removal of the sample before gelation makes the protection of the refractometer prisms by film covers unnecessary. On the other hand, a preliminary study of the system in question by the elaborate method will be useful to determine the proper time limits.

This method has been worked out for the system Epon 828 + 14 phr m-phenylenediamine at 65°C. The results of the preliminary study are presented in Figure 11. It was concluded that the best time for the first reading was at 10 minutes after mixing and the proper time for the second reading at 70 minutes after mixing. Since gelation occurs approximately 110 minutes after mixing, there is an ample margin of safety. The result is expressed by the difference of the readings, namely $(n_{70\ min}^{65} - n_{10\ min}^{65})\ 10^4$. This value is called "Refractometric Cure Rate". The reproducibility of the results is within ± 2 units. A complete description of the simplified method is given in the appendix.

Conclusion

Refractometry can be recommended as a tool for determining cure rates and conversions of epoxy resins. A somewhat elaborate method is suggested where measurements from inception to completion of the cure are required or when the behavior of the sample is not thoroughly known. A sim-

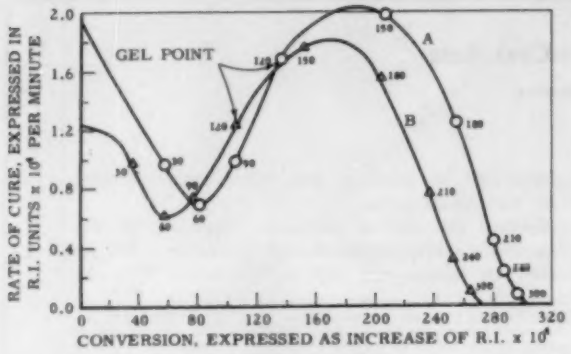

Figure 8. Cure rates with a secondary amine in the presence of phenolic hydroxyl.
A. Epon 828 + 5 PHR piperidine + 5 PHR phenol. B. Epon 828 +5 PHR piperidine + 5 PHR bisphenol-A. Numbers on curves indicate cure time in minutes.

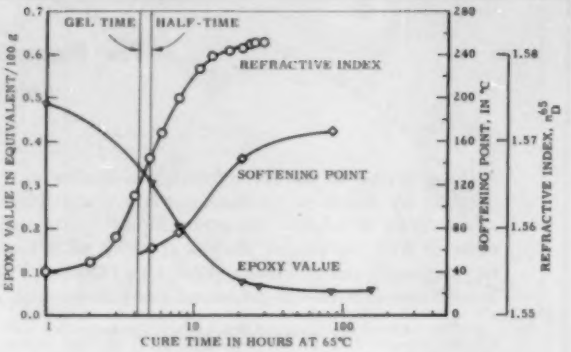

Figure 10. Cure of Epon 828 with 5 PHR piperidine.

plified method is recommended for routine work such as quality tests on epoxy resins and curing agents.

Acknowledgement

The author wishes to express his thanks to O. D. Bergren and R. H. Fleming for their assistance in the experimental part.

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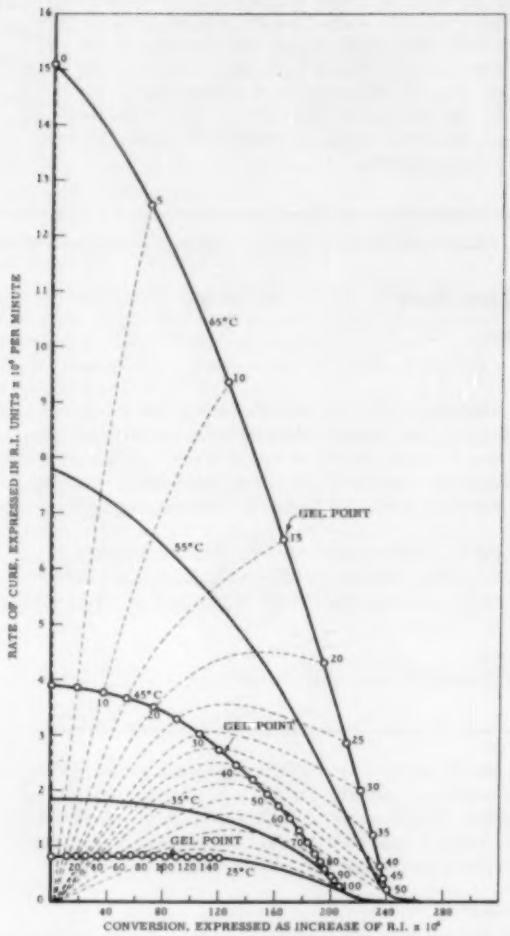

Figure 9. Cure rates of Epon 828 with 5 PHR ethylenediamine at different temperatures.
Broken lines connect points of equal cure time. Numbers on curves indicate cure time in minutes.

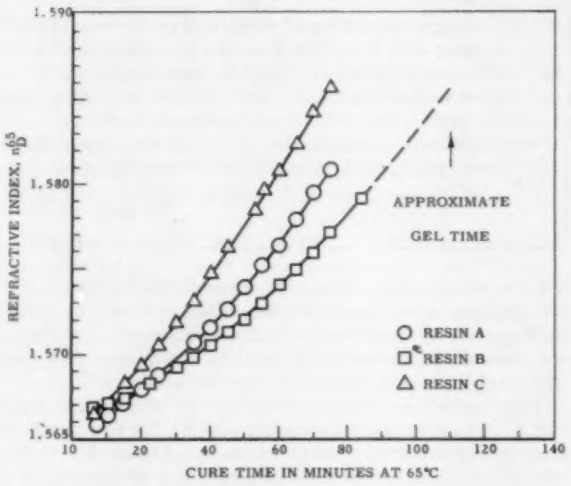

Figure 11. Cure rate of different epoxy resins with 14 PHR of m-phenylenediamine.

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Plastic Radomes at Elevated Temperatures Theory and Practice

Possibility for the future: Ceramic-fiber-wound-roving type of reinforced inorganic laminate structure for plastic radomes

With the rapid upward trend in speeds of manned aircraft, guided missiles, and even satellite-launching vehicles, operation of radomes at elevated temperatures has become a necessary practice. The resultant sharpening of both government and industrial interest in the possible uses of ceramics for radomes makes it particularly appropriate that we reexamine and reappraise the position of high-temperature plastics in this field.

Since elevated temperature is the big obstacle to the use of reinforced plastics in radomes and thermal-shock the chief drawback to the use of ceramics, the nature of the probable modes of flight operation should be examined. Two modes—long duration flight at subsonic and transonic speeds, and long duration flights at lower supersonic speeds—do not pose too great a problem for reinforced plastics. Therefore, the four newer modes of intended flight will be discussed below. The graphs below were plotted from data developed from calculations based on the methods of Gatewood (1) and Hill (2). Similarly the temperature gradients built up across the radome-wall section were deduced from the preceding data and used to calculate the critical stresses attributable to thermal-shock for the competing ceramic radome cases. The methods of Gatewood (1) and Hill (2) were used here also. For this purpose, the simpler, but overly pessimistic condition of complete physical restraint of the analysis model was assumed. The resultant thermal-stress figures then were reduced to stress-to-weight ratio form and compared graphically with the strength-to-weight ratio figures for the ceramic material. Thereafter, even a cursory inspection of the resultant thermal-shock criteria, superimposed on Figures 1, 2, 3, and 4, clearly demonstrates the critical nature of thermal-shock in the competitive brittle ceramic constructions.

MATERIALS OUTLOOK

IF . . .

upper limits of stable operation can be pushed above 600°F, use of reinforced plastics on long-duration supersonic aircraft is possible. Plastics, however, will have to contend with competition from the more stable ceramics.

AND . . .

the most likely usage for plastics in hypersonic flight is apt to be as a non-carbonizing, non-stress-bearing ablative coating, applied to the outer surface of a ceramic radome.

BUT . . .

plastics will probably take over in the ballistic-reentry situation. Reason: The short-duration of this extreme exposure favors the ablation characteristic of plastics.

FOR THE FUTURE, THOUGH . . .

the best approach seems to be the development of inorganic laminates of heat-stable ceramic reinforcing fibers, coupled with some form of compatible, tough, inorganic-impregnating cement or other bonding matrix.

Long-duration Flight at the Lower Supersonic Speeds (more than 20 minutes at speeds of Mach 3.5 or less).

Referring to the lower curve of Fig. 1, it is noted that the outer-skin surface-temperatures rise at a modest rate, passing the conventional steady-state limit in approximately 30 seconds. As flight continues, the outer-surface temperature continues to rise toward a crest temperature of about 700°F, until modified by incipient ablation. It is noteworthy that the inner-surface temperatures rise more slowly and approach a somewhat lower crest temperature determined by the degree of inner-surface cooling provided. This curve then also serves to justify the previously acknowledged acceptability of operating low-temperature plastics in short-duration flights of outer-surface temperatures which are well above steady-state operating limits. On the other hand, reference to the upper curve of Fig. 2 shows that both the inner and outer surface temperatures rise almost in unison

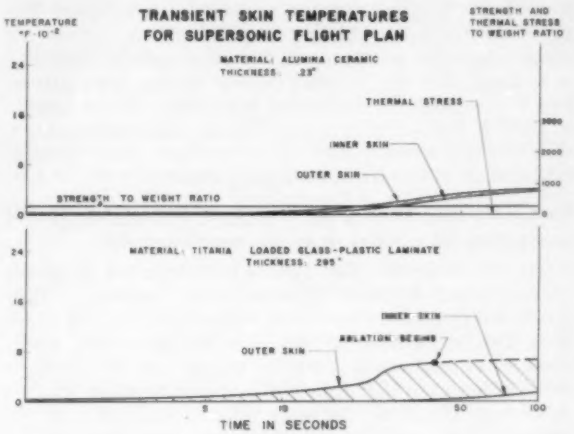

Figure 1

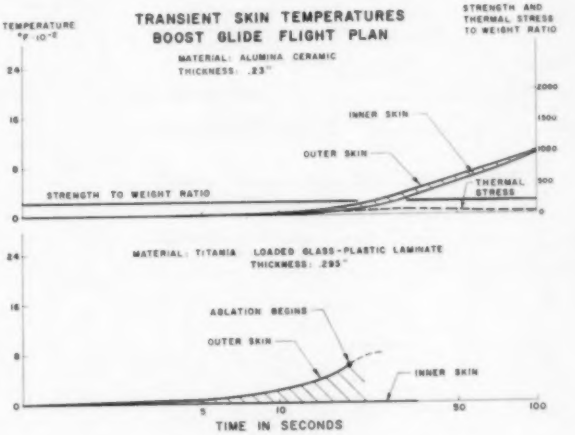

Figure 2

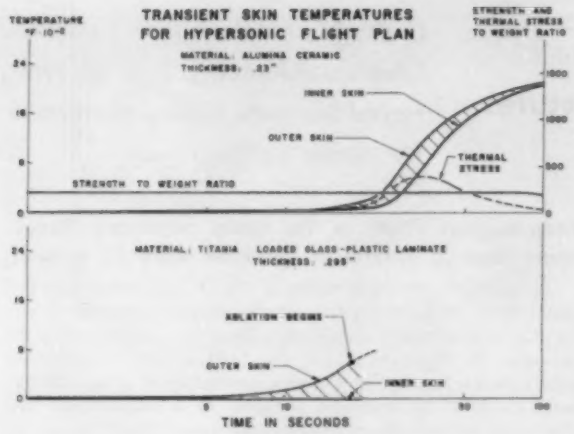

Figure 3

at a mild rate toward a crest temperature in the vicinity of 520°F. Only negligible thermal-stress is built up in the wall.

Short-duration Flight at Hypersonic Speeds (up to 20 minutes at speeds near Mach 6).

Reference to the lower curve of Fig. 2 shows that the outer-surface temperatures rise rapidly toward a theoretical crest temperature of 2000°F, until modified by ablation (after about 20 seconds). This rate-of-rise is so rapid that the outer surface is ablating long before any appreciable temperature-change occurs at the inner surface. Similarly, it is noted that the ceramic radome surface temperatures rise very rapidly toward a crest temperature in the vicinity of 2000°F. But, while even the ultimate crest temperature is well within the steady-state operating limits of the ceramic, the build-up of thermal-stress is dangerous.

Long-duration, Boost-glide Flight at Hypersonic Speeds (more than 20 minutes at speeds near Mach 10).

The lower curve of Fig. 3 traces a situation for the plastic radome which is almost identical to the hypersonic flight mode. Only the theoretical crest temperature, toward which it is directed, differs. In this case the crest temperature reaches approximately 3000°F instead of the previous 2000°F but even this makes little difference, since ablation takes over at roughly the same point in each case. On the other hand, the ceramic radome-surface temperature curves (both outer and inner) rise almost coincidentally at a modest rate toward a crest temperature near 3000°F. Only

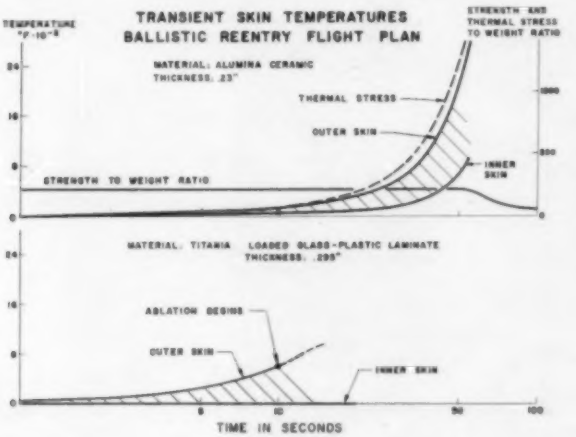

Figure 4

a modest peak of thermal-stress is built up about 40 seconds after commencement of the boost-glide flight mode. Its peak is well below the radome's static strength. No appreciable strength reductions from thermal causes are noted within the 100-second transient period examined, but some strength reduction at the crest temperature reached later in the flight is expected.

Short-duration Ballistic Reentry at Hypersonic Speed (in excess of Mach 12).

Inspection of the lower curve of Fig. 4 again exhibits a very similar situation to that obtaining for plastic radomes in the preceding hypersonic and boost-glide flight modes. The rise in temperature begins earlier and thereafter proceeds in a somewhat more linear fashion until the onset of ablation modifies it. The total expected duration of the heating cycle, however, is much shorter than those of the preceding two flight modes, thereby making the situation more favorable to usage of plastics. In contrast to this, the upper curve of Fig. 5 exhibits a catastrophic rate-of-rise of both ceramic-surface temperatures and thermal stess, which far exceed the indicated limits of the ceramic material, at least in its unprotected form.

Dielectric Stability Considerations

As indicated in earlier paragraphs of this paper, there is another major factor to be considered. While physical integrity must be given first concern, practical operation also demands stability in the electro-optical performance of the radome. Prerequisite to practical design for such stability is the selection of materials whose dielectric constant and loss tangent remain reasonably stable with varying temperature. Obviously, there is no point in working out a carefully corrected guidance radome design for room temperature if that design performance is lost immediately on rising to operational temperatures. This is particularly true when one recognizes that operations' temperatures vary with every change of trajectory, and even vary from point to point over the radome.

Thus, it is seen that a stable dielectric constant is particularly essential to those critical elevated-temperature radome applications in support of precision guidance radar usage. An idealized target figure for such stability is that dielectric constant of a plastic should vary no more than 2% over the operating temperature range. A corresponding figure for ceramics would be 3%. Actually, thickness changes, associated with temperature changes, affect these target figures also. Hence, an idealized objective would call for a compensatory decrease of dielectric constant for any tendency of the material to expand with temperature. The reverse would be true for contraction of the material with temperature change.

Conclusions

Whether plastics win out over ceramics, for supersonic and hypersonic missiles and aircraft, will depend upon the vigor with which development effort is pushed in each area. There is realistic potential evident in both fields, but the greatest potential appears to lie in a merging of the two approaches into some form of tough ceramic-fiber wound-roving type of reinforced inorganic laminate structure.

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Interfacial Properties of Polyesters at Glass and Water Surfaces [1]

One of the principal problems in the construction of glass-fiber reinforced plastics is the adhesion between the resin and glass fiber. Failures in reinforced plastics appear to occur in many cases at the resin-glass interface, that is, in adhesive failure. Therefore, an investigation has been made of some of the fundamental properties of adhesion applicable to resin-glass fiber systems in reinforced plastics.

Interfacial studies have indicated correlations between experimental data and actual performance. Glazer (6) conducted monolayer studies on epoxy type molecules and their derivatives and found by comparison of film collapse pressures that the hydroxyl group exhibited superior adhesive properties towards aqueous subphases. Studies by deBruyne (2) on several epoxy resins derived from bisphenol and resorcinol indicated correlation between the adhesive action of these resins and their hydroxyl group content. Some possible effects of chemical composition and structure on adhesion were indicated by the adsorption studies of Murphy and Page (13). The work of Jenckel and Rumbach (9), and of Koral, Ullman and Eirich (11) indicates that adsorbed polymer is attached to the solid surface in a coiled configuration, with portions of the molecule extending away from the solid surface as bridges.

In this article two approaches to the investigation of interfacial relationships are reported. One is a study of the behavior of stearic acid and polyester monolayers on aqueous subphases, carried out with a Langmuir film balance. The other is a study of the adsorption of polyester resins on glass, silica, and alumina. A more detailed report on the polyester adsorption and a comparison with other systems have been previously made (15).

Monolayer Studies

A very small quantity of an insoluble, non-volatile organic material, such as an oil, may or may not spread to form a film when placed on the surface of a liquid of high surface tension or free surface energy such as water. The ability of the oil to spread is determined by free energy changes accompanying the process. Fortunately, from a practical standpoint, the capacity of the oil to spread on a water surface can be expressed in terms of the work of adhesion, Wa, and cohesion, Wc. These energy terms may be used to define the spreading coefficient, S (7, 8). The definition of S is

$$(1) \quad S = -\left(\frac{\partial F}{\partial A}\right)_{P,T} = Wa - Wc,$$

where F is the free energy of the system, A the available area of the subphase, Wa the work of adhesion between the oil and water, and Wc the work of cohesion of the oil. The spontaneous spreading of the oil to form a film requires a decrease in the free energy of the system. As shown in Equation (1), this energy decrease can occur only if S is positive or the work of adhesion is greater than the work of cohesion. If the oil spreads, the film existing after mutual saturation of the oil and water phases is usually a monolayer.

If the energy associated with a surface is considered to be the surface tension, the difference between the surface tension of the clean and monolayer-covered water may be defined as the surface pressure, π (1, 7). The expression for π is

$$(2) \quad \pi = \gamma_w - \gamma_f$$

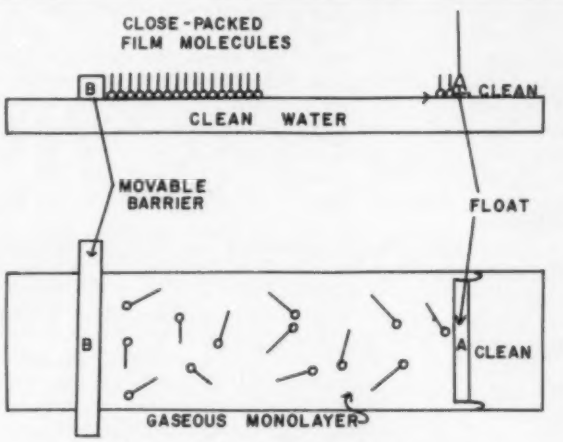

Figure 1. Horizontal film balance.

[1] This work was conducted, in part, under a project sponsored by the Bureau of Aeronautics, Department of the Navy.

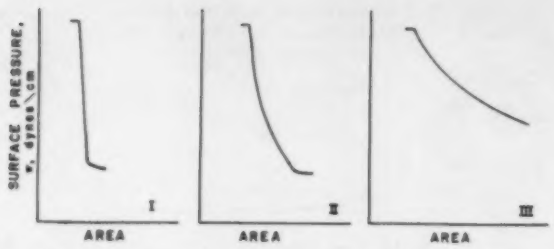

Figure 2. Typical surface pressure-area isotherms of monolayer films.

Classification of Monolayer Film Isotherms

Intermolecular Cohesion	Type of Curve	Isotherm
High	Condensed or Solid	I
Medium	Liquid-Expanded	II
Low	Gaseous	III

where γ_w is the surface tension of the pure water and γ_f that of the water covered with a monolayer of oil.

Many polar organic molecules spread to form monolayers on aqueous subphases. The spreading characteristic of these molecules permits the use of surface film techniques for investigating the interfacial properties of simple and polymeric organic molecules. Some of the properties that may be studied are the molecular size, relative adhesive and cohesive forces, and molecular orientation.

The interfacial characteristics of monolayers are usually studied with the surface film balance. Figure 1 shows diagrammatically the horizontal film balance. The essential parts of the apparatus are a large tray that contains the subphase liquid, a movable barrier to compress the monolayer film, and a sensitive device, the float, to measure small changes in the surface pressure. The data collected are graphically represented as surface pressure-area, π-A, isotherms. These isotherms are used to determine the interfacial properties of simple and polymeric organic molecules.

Before examining the experimental isotherms, the main characteristics of π-A isotherms and their relation to molecular properties will be discussed (1). In many cases the molecules of the monolayer are arranged in a simple manner, and the interfacial characteristics of the molecules are relatively simple. For example, the two-dimensional monolayer may have properties that are similar in many respects to one or all of the principal states of matter in three dimensions. The fundamental types of monolayer isotherms commonly observed are shown schematically in Figure 2. Isotherm I is obtained from condensed or solid films where the molecules are closely packed and the intermolecular cohesion is high. Isotherm II demonstrates the type obtained from liquid type films. These films are still coherent but occupy relatively large surface areas. Isotherm III is representative of gaseous type films where the molecules are separate and move about independently on the surface. These film types are best classified according to the lateral cohesion between the molecules. However, the stability of the monolayer to lateral compression is determined by the combined effects of intermolecular cohesion and the attraction or bonding between the film molecules and the subphase.

A horizontal surface film balance was used to investigate the monolayer properties of several simple and polymeric organic molecules of the type shown in Figure 3. A portion of the data obained is graphically represented as surface pressure-area isotherms in Figures 4, 5, 6.

The isotherm in Figure 4 shows the interfacial characteristics of a stearic acid monolayer on distilled water. This isotherm is of the condensed type, except at very low surface pressures, indicating that the intermolecular cohesion is high. The limiting area of 24·9A² per molecule was obtained by extrapolating from the region of low compressibility to zero surface pressure. This value is approximately the area occupied by the carboxyl group. Thus, the molecules are rather closely packed. Unbranched long chain acids packed in this manner have their hydrocarbon chains inclined to the subphase surface since the molecules are anchored in the subphase by the polar end group, — COOH. If, however, the stearic acid is spread as a monolayer on a M/2000 $CaCl_2$ subphase, the monolayer characteristics (Figure 5) are not the same as on distilled water. At surface pressures below 0.5 dynes/cm, the monolayer is

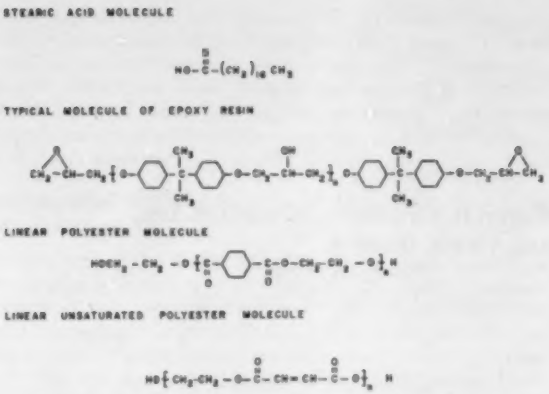

Figure 3. Organic molecules with polar groups.

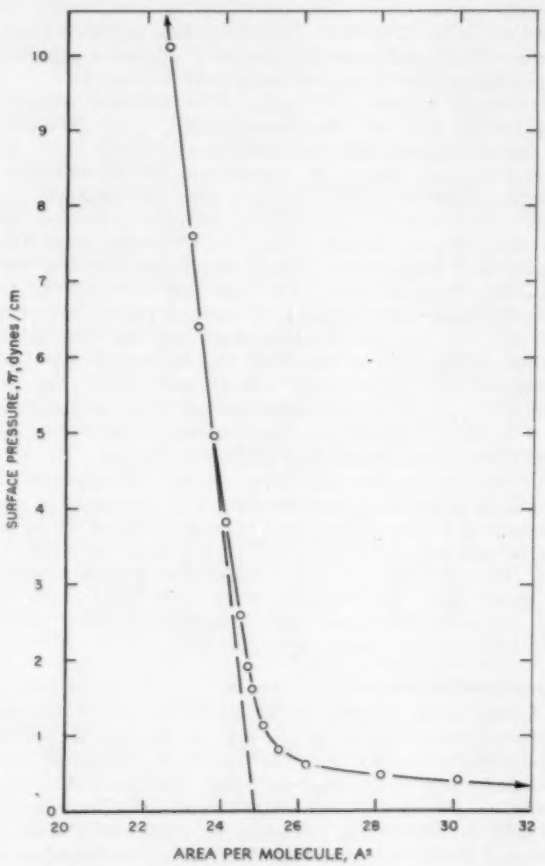

Figure 4. Surface pressure-area isotherm of stearic acid on distilled water at 25.0 ± 1.0°C.

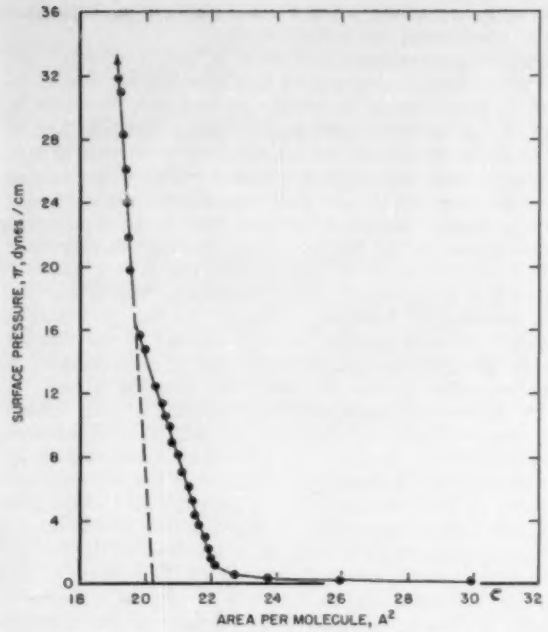

Figure 5. Surface pressure-area isotherm of stearic acid on calcium chloride solution at 25.0 ± 1.0°C.

Concentration of $CaCl_2 = \frac{1}{200}$ mole

pH of $CaCl_2$ solution = 5

of the expanded type, but at pressures between 2-16 dynes/cm the film is of the condensed type. For surface pressures above 16 dynes/cm the monolayer exists as a solid film. The limiting area per molecule for a molecule in the solid film is 20·3A². This value agrees very well with the theoretical value of 20·5A² (10, 16) for a closely packed stearic acid monolayer. In the solid film the hydrocarbon chains of the molecules are nearly perpendicular to the subphase surface.

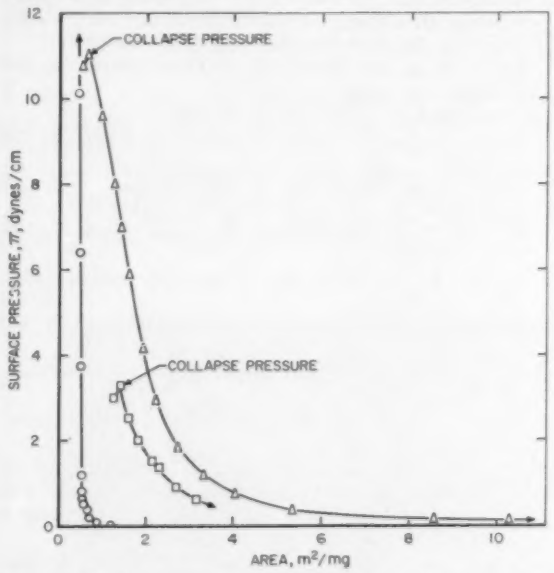

Figure 6. Surface pressure-area isotherms of polar organic molecules on water at 25.0 ± 0.5°C.

○ Stearic Acid
△ Poly(ethylene adipate)
□ Poly(ethylene succinate)

The monolayer properties of linear saturated polyesters are quite unlike those of the straight chain organic acids. The monolayer characteristics of two linear saturated polyesters, differing chemically by two methylene units per segment, poly(ethylene succinate) and poly(ethylene adipate) are compared to each other and to stearic acid on distilled water subphases in Figure 6. For these isotherms the area axis is expressed as m²/mg since the exact molecular weights of the polyesters were not known. The isotherms show that the polyester molecules are more compressible than those of the organic acid. In addition, it is interesting to note the markedly different monolayer properties of the two polyesters. At surface pressures below 0.4 dynes/cm the poly(ethylene adipate) monolayer is the gaseous type, but at higher pressure is transformed into the liquid expanded state. On the other hand, poly(ethylene succinate) remains in a highly expanded state up to monolayer collapse. At very low pressures the stearic acid monolayer is the expanded type, but transforms rapidly into the condensed state at higher surface pressures.

Adsorption Studies

Linear saturated polyesters of different chemical composition, with number average molecular weights of the order of 4000, were adsorbed onto glass, silica, and alumina. The study was confined to adsorption from dilute solutions, where polymer adsorption has received theoretical treatment by Simha, Frisch, and Eirich (3, 4, 5, 14).

The glass adsorbent was Type-E glass, the type most commonly used in glass-fiber-reinforced plastics. Glass marbles were ground in a ball mill to pass through a U. S. Standard Screen No. 325 (44 micron holes). The resulting powder was nonporous and had a Brunauer, Emmett, Teller (B.E.T.) surface area of 0.68 m²/g. The silica was a type used as a reinforcing agent for rubber (Cab-O-Sil) with an average particle diameter of 15 to 20 millimicrons and a B.E.T. surface area of 190 m²/g. It also was nonporous. The alumina was a porous material with a B.E.T. surface area of 210 m²/g.

The procedure usually followed was to add a polymer solution to the adsorbent, seal the tube, and rotate the mixture in a constant temperature bath for approximately 20 hours. After adsorption the mixture was centrifuged and the concentration of polymer was determined by measurements of the carbonyl absorption band in the vicinity of 5.8 microns.

The curves shown in Figure 7 show the adsorption of one of the polyesters, poly(neopentyl succinate), on Type-E glass under different conditions. The polymer adsorbed per gram of glass powder is plotted against the final solution concentration. There is considerably more polymer adsorbed from the poor solvent, toluene, than from the good solvent, chloroform. This effect was observed with all the polyesters investigated. The adsorbed molecule appears to be tightly bound to the surface, as shown by the irreversibility of the adsorption, both from the toluene and chloroform solutions. Increasing the temperature at which absorption took place from 30° to 86.8°C brought about a decrease in the amount of polymer that was adsorbed from toluene solutions. However, no difference in the amount adsorbed could be measured for the temperature range 30°-50°C for adsorption from chloroform solution.

Inasmuch as the polymer molecule most probably exists as a more open structure in the good solvent than in the poor, it is reasonable that each adsorbed molecule would occupy more surface area when adsorption is carried out from a good solvent than when carried out from a poor solvent. This may account for the larger quantity adsorbed from the toluene solution. In addition, there is an increase in the solubility and probably in radius of the polymer mole-

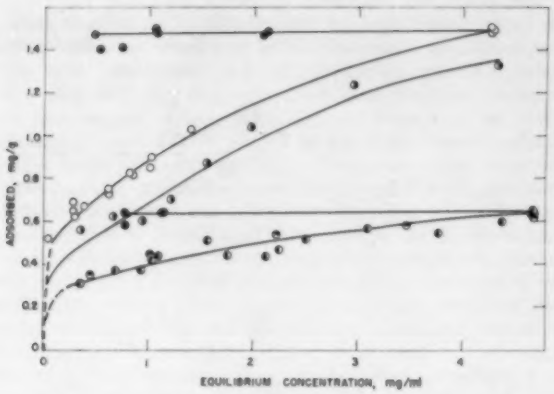

Figure 7. Adsorption of poly(neopentyl succinate) onto type-E glass.
O Adsorption from toluene at 30°C
Circle, bottom half solid: Desorption at 30°C with toluene
Circle, right half solid: Adsorption from toluene at 86.8°C
Circle, left half solid: Adsorption from chloroform at 30°C
● Desorption at 30°C with chloroform
Circle, top half solid: Adsorption from chloroform at 50°C

cule when the temperature of the toluene solution is changed from 30° to 86.8°C, bringing about a decrease in the polymer adsorbed. As the molecule is already in an extended condition in the chloroform solution, an increase in temperature from 30° to 50°C probably does not appreciably affect the polymer radius or solubility, resulting in no measurable change in the adsorption isotherm.

The amount of poly(neopentyl succinate) adsorbed per unit surface area is plotted in Figure 8 against the equilibrium solution concentration for glass, silica, and alumina. The adsorptions were carried out from chloroform solutions. The largest quantities were adsorbed on the glass and the smallest on the alumina. The small amount adsorbed on alumina may be a result of the porous nature of this material and the inaccessibility of portions of its internal surface area to the bulky polymer molecules. As both the Type-E glass powder and the silica were nonporous, it appears that the components of the glass, in addition to silicon dioxide, present additional sites for the attachment of the resin molecules.

Discussion and Conclusions

The two techniques described here indicate the probable configuration of small polyester molecules at the glass surface and the manner by which they are attached. It was shown in Figure 7 that the quantity of polymer adsorbed per gram of adsorbent is greater from a poor solvent, toluene, than from a good solvent, chloroform. Assuming total surface coverage, the number of surface attachments per resin molecule is different in the two cases. The lack of desorption with decreasing solution concentration indicates that these attachments are rather strong.

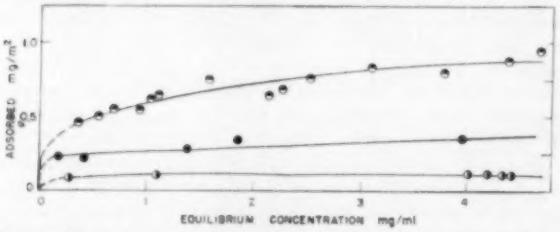

Figure 8. Adsorption of poly(neopentyl succinate) from chloroform solution onto several adsorbents.
Circle, top half solid: Adsorption onto Type-E glass
● Adsorption onto silica
Circle, right half solid: Adsorption onto alumina

Surface pressure-area isotherms of poly(neopentyl succinate) on distilled water show that a monolayer film of the resin in a condensed or closely packed state occupies an area of 2.10 m^2/g or 0.48 mg/m^2 (12). The molecules lie flat on the water surface and are attached by the ester groups. It can be seen from Figure 8 that a sufficient quantity of this resin is adsorbed onto glass from chloroform solution to form a closely packed monolayer film at an equilibrium concentration of 0.5 mg/ml. At an equilibrium concentration of approximately 5 mg/ml, 0.85 mg/m^2 are adsorbed from this solvent. This would correspond to slightly less than two monolayers. Assuming attraction to the glass surface through the ester groups, the total amount of polymer adsorbed at this higher concentration can be accounted for by some distortion of the molecule away from the glass.

In the case of adsorption from toluene, a poor solvent, the amount of polymer adsorbed onto glass at an equilibrium concentration of about 5 mg/ml would correspond to approximately 4 to 5 monolayers. The curve has not reached a plateau at this concentration (Figure 7) and even more polymer can be deposited. It appears that in this case a smaller portion of each molecule is attached to the surface. The work of Koral, Ullman, and Eirich (11) with poly(vinyl acetate) indicates that in the case of very high molecular weight polymers, relatively large portions of the chain extend into the solution.

In conclusion, it appears that the configuration of an adsorbed polymer molecule and the number of attachments per molecule on a surface such as glass are variable and probably molecular weight dependent. This can vary from what appears to be a rather flat configuration for the small polyester molecules from a good solvent to what are probably extended, inter-associated loops for the large polymer molecules. It is obvious that a more detailed knowledge of the interfacial properties described here and a measurement of the thickness of the adsorbed polymer film will be required to accurately evaluate the resin-solid interface.

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NATIONAL RUBBER MACHINERY CO.
PLASTICS EQUIPMENT DIV.

Speaking of Extrusion

Vacuum Feed Extrusion

An answer to the problem of entrapped air and other gases in the extrudate.

Since the earliest days of thermoplastics extrusion, it has been a common practice to feed the plastic material, in whatever form available, to the extruder feedscrew at atmospheric pressure. Extrudate porosity, inadequate dispersion, and other extrusion problems have been remedied by preheating of the material, changing feedscrew characteristics, valving, etc. Recent investigation indicates a new process—described as vacuum feed extrusion—may help eliminate some of these annoying problems.

Thermoplastic vacuum feed extrusion is essentially a process which incorporates a system and equipment for the extrusion of thermoplastic materials so that a nonporous, noncellular, or controlled cellular product may be extruded from solid thermoplastic materials in powder, granule, chip, pellet, or like form. This process involves the feeding and melting of the thermoplastic material within an extruder which is modified to allow for a controlled vacuum atmosphere within the system.

The following equipment is required to convert a conventional plastics extruder so that it can be used for vacuum feed extrusion:

1. An air-tight container (vacuum feed hopper) fitted with the openings and controls necessary to control the vacuum and material feed, either manually or automatically. This vacuum feed hopper is fastened to the extruder feed opening and sealed to prevent air leaks.
2. An air-tight seal between the cylinder barrel and the feedscrew bearing section.
3. A vacuum pump with adequate capacity to maintain a desired vacuum setting.

The most apparent improvement derived from vacuum feed extrusion is the removal of entrapped air or gases. Tests indicate the system is effective with all types of thermoplastic compounds varying from highly loaded to near pure resin.

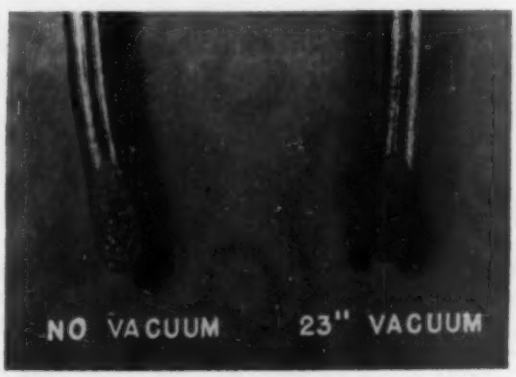
Case history No. 1 shows the effect of vacuum extrusion on a vinyl dry blend wire covering compound which was difficult to properly extrude.

Case History No. 1—Extrusion Information

Material: Plasticized Vinyl Dry Blend—Wire Covering Compound
Extruder: NRM Induction Heated, 2.5″ Diameter, 20:1 L/D
Die: ¼″ Diameter Rod
Feedscrew: Deep, Full Flighted, 2 to 1 Channel Volume Ratio
Screens: One 20-Mesh and One 40-Mesh
Screw Speed: 38 R.P.M.
Feedscrew Temperature: Ambient
Stock Temperature: 340°F.

With no vacuum being applied to the feed system, the extrudate rod was very porous. With 23″ Hg. vacuum applied to the feed system, the extrudate rod was completely homogeneous. Output was approximately 98 #/hr.

Case history No. 2 shows the same improvement on a non-loaded, clear PVC film compound.



Case History No. 2—Extrusion Information

Material: PVC Film Compound
Extruder: NRM Induction Heated, 2.5″ Diameter, 20:1 L/D
Die: ¼″ x 3″ Strip
Feedscrew: Shallow, Full Flighted 3.5 to 1 Channel Volume Ratio
Screens: One 20, One 40, and One 60-Mesh
Screw Speed: 50 R.P.M.
Feedscrew Temperature: Ambient
Stock Temperature: 375°F.
Output: 90 #/hr.
Gage Pressure at Screen Pack: 900 P.S.I.

The photo shows the uniform homogeneous extrudate which was produced with a vacuum of 25″ Hg. In contrast, the extrudate sample made with no vacuum was of poor quality and filled with bubbles. Without vacuum, increasing the screen pack density and introducing cooling water to the feedscrew improved the extrudate quality some, but at the expense of reduced output.

Recent tests on a high speed 2.5″ NRM extruder have produced outputs of over 300 #/hr. at about 200 r p m vacuum feeding a plasticized vinyl dry blend.

Case History No. 2–Continued

Photo below shows considerable improvement in dispersion resulting from vacuum feed extrusion. Because the material is compacted immediately in the feedscrew, the effective length of the extruder is increased. However, there is an even more important improvement which results from vacuum feed extrusion. In normal extrusion, air or gas entrapment make it difficult to apply uniform mechanical working to the plastic particles surrounding the voids. By eliminating the voids, all particles are subjected to the same amount of mixing action, thus aiding the dispersion process.

Case History No. 3—Extrusion Information

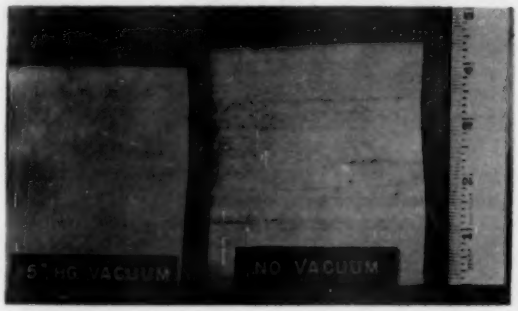
Material: Plasticized Vinyl Dry Blend
Extruder: NRM Resistance Heated, 3.5" Diameter, 20 to 1 L/D
Die: ⅜" x 4" Strip
Feedscrew: Shallow, Full Flighted, 3.8 to 1 Channel Depth Ratio
Screens: One 20-Mesh and One 40-Mesh
Screw Speed: 78 R.P.M.
Feedscrew Temperature: Ambient
Stock Temperature: 340°F.
Gage Pressure at Screen Pack: 800 P.S.I.
Output: 450 #/hr.

Normal extrusion conditions produced an extrudate strip exhibiting poor carbon black dispersion, rough surface finish, and some porosity. Applying a vacuum of 25" Hg. to the feed system improved the surface finish, dispersion, and removed the voids.

The limiting factor in the extrusion of hi-impact polystyrene sheet at high output rates has been the reduction in quality as the feedscrew speeds are increased. The imperfections may appear as small pock marks, streaks, and bubbles. They are usually caused by entrapped air, or small amounts of moisture which are not removed from the material. Tests have shown (case history No. 4) that vacuum feed extrusion of good quailty hi-impact polystyrene sheet can be achieved at output rates much higher than can normally be produced under normal atmospheric feed conditions.

Case History No. 4—Extrusion Conditions

Material: Hi-Impact Polystyrene
Extruder: NRM Resistance Heated 3.5" Diameter, 20:1 L/D
Die: NRM 42" Manifold
Feedscrew: Dulmage
Screens: 20, 40, and 60-Mesh
Screw Speed: 72 R.P.M.
Feedscrew Temperature: Ambient
Stock Temperature: 375°F.
Output: 281 #/hr.
Gage Pressure at Screen Pack: 900 P.S.I.



Conclusions

Thermoplastic vacuum feed extrusion is a relatively new approach to solving some of the problems which are present in the thermoplastics extrusion industry. The industry should look upon this process and the related equipment as a new tool to assist them in producing a better product at a lower cost.

This process should not be confused or directly compared with the vented extruder. In the thermoplastic vacuum feed extrusion process the air, and heat released gases, and small amounts of surface volatiles are removed before or as soon as the thermoplastic begins to melt; whereas, in a vented extruder the thermoplastic material is completely molten before it is delivered to the venting section.

It has been found that the following advantages can be gained from the use of the thermoplastic vacuum feed extrusion process:

1. Removal of air, heat released gases, and small amounts of moisture.
2. Less extrudate swell at the die.
3. Under some conditions the degradation of material within the extruder and die can be reduced.
4. Higher feedscrew speeds and greater output.
5. Predrying of material not as critical (in some cases it may be eliminated).
6. Reduced surging.
7. Powder materials and dry blends are densified and feed better.
8. Better dispersion of solids and additives.

edited by **Bruce H. Maddock**, *Union Carbide Plastics Co.*

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Rigid vinyl pipe from dryblend	170
Polyethylene on cellophane	170
3½" (40 H.P.)	
Vinyl garden hose	480
Polyethylene pipe	380
Roll cast polypropylene film	370
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High impact polystyrene sheet, vented	670
Cellulose butyrate sheet, vented	640
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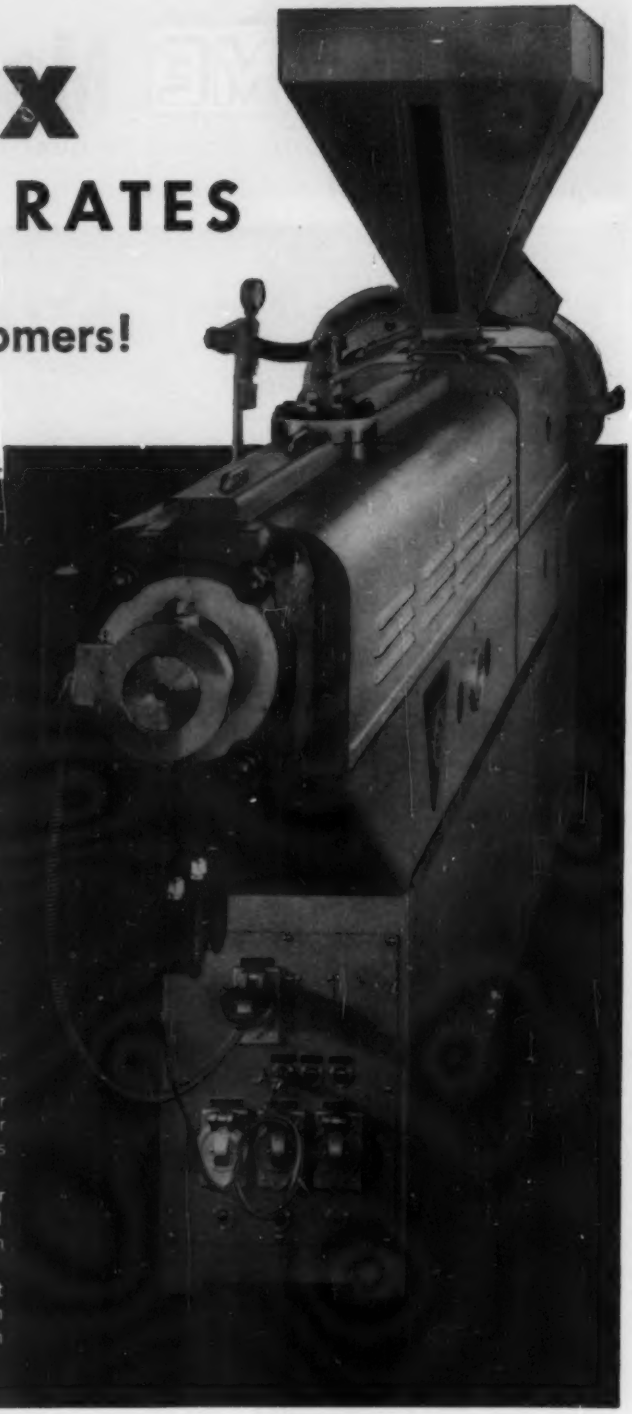

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59-A

Moldmaking

GEORGE E. PICKERING, AIR FORMED PRODUCTS CORP.

MOLDS FOR BLOW MOLDING

Thinking of going into blow molding? Here are some facts which will help you get started

Blow molds can be produced from many different metals using more or less conventional metal processing equipment. The usual problems of cooling, machining and assembly difficulties, weight and durability of the finished blow mold are as common as they are with conventional molds.

How They're Made

Probably the most popular process used for blow molds at present is casting—both atmospheric and pressure casting. Almost any shape can be produced by the casting process. The process is, therefore, undoubtedly the most versatile and reproductive one available. The resulting mold, however, is not always the most economical, versatile or durable.

Atmospheric casting has the advantage of low equipment cost. Some is being accomplished on a captive basis. It has the disadvantages of porosity and lack of toughness in the material. This, in turn, may make insert work difficult to accomplish. Appearance details such as surface texture, engraving, etc. are more uniform from cavity to cavity than can be accomplished by engraving on machined cavities. Atmospheric casting has severe limitations as to the details that can be cast. This has led to the development of pressure cast cavities.

Pressure casting is probably the fastest growing process in use today for blow molds. It has been used commercially with all five of the metals mentioned below and experimentally with many other metals and alloys.

Turning is excellent for a small number of small round dies. It permits the economic use of steel with its inherent toughness allowing the use of interchangeable inserts for maximum flexibility. The high specific gravity is usually no problem on small items.

Duplicating could be considered a "tried and true" process for blow molds. It has strongly in its favor—availability and predictability. On "crash programs" it can frequently be worthwhile to accept a slightly higher cost rather than risk a possible delay.

Press hobbing has generally been too expensive a process for blow molds. It would be of value mainly where hardened steel is desired in the entire mold. Such a condition might arise on long runs using multiple cavities under hard usage. Processes having extremely fast closing actions or closing under injection molding type pressures, as used in the "mold and blow process", may justify the added expense of hardening.

Molds for Blow Molding and Injection Molding— Here are the Differences

1. Blow molds use cavities only—this eliminates matching force plugs with cavities.

2. Blow molds seldom, if ever, require hardening—a decided economic advantage.

3. Material movement within a blow mold is a stretching rather than a flowing action—this reduces problem of weld and flow lines, mold erosion, trapped gas, etc.

4. Undercuts on blow molded items can be stripped more easily than injection molded pieces—eliminates need for expensive sliding inserts, levers, cams, etc.

The Materials

Currently, most of the commercial blow molds have been made of the following metals: steel, aluminum and alloys, beryllium-copper, cast iron and cast zinc alloys. Metal spraying and electrolytic deposition methods have been utilized to a limited extent. For the present, these are more or less experimental but should be watched carefully in the immediate future.

Some work has also been done in making blow molds of cast plastic and cast plastic metal mixtures and with coating of these and other materials on conventional metal molds. These materials have a serious handicap in low heat transfer but have the promise of low cost and speed of production. Their greatest potential: quick sample molds.

Design Factors

Of the multitude of factors affecting the design or construction of blow molds, the most influential is the design of the item to be blown. Exaggerated size relationships such as extremely narrow or extremely wide tops, bottoms or mid-points are not only difficult to blow but the difficulty is frequently reflected and sometimes magnified in the various types of die construction.

If an item is basically round and smooth in cross section it can usually be turned on a lathe. Template turning is needed for return curves.

The smaller the item and the fewer cavities required, the more reason to use turning to produce the mold, particularly if high quality is wanted or the possibility of interchangeable inserts or bottom plates is involved.

For turned molds, inserts in either or in both top and bottom are required in order to gain entrance for the tool in turning. Inserts, both top and bottom, are advisable wherever

high specifications such as snap fits, threads and push-up bottoms are required or when interchangeability is desired.

When several round dies with no inserts are involved casting might work out cheaper. Hobbing may be of value here but may be more expensive in the long run unless a hardened die is a requirement.

Multiple cavity round dies favor casting, particularly if dies are side by side and a one-piece construction is feasible. One-piece construction avoids the problem of heat loss at the interface of insert. Variables can be accomplished in casting by incorporating inserts in the original hobs and making separate dies to each variable.

Any shape that cannot be turned is considered irregular and is usually duplicated or cast. Metal spray and electrolytic deposition are also possible but have not been widely used commercially.

When a product design is frozen to a single style, with no variations, the mold making problem is greatly simplified and strongly favors casting. In many cases, however, it is desired to have a variety of style changes incorporated in a single die. Engineers deplore this arrangement but economics often dictate the end result.

A classic example is a cylinder bottle with interchangeable neck inserts and bottom plates affixed to each end of a straight cylinder. In this case, machining is usually the most economical method of construction in the long run, using steel throughout to resist the wear and tear from repeated changes in inserts. For irregular shapes, duplicating in steel would be advisable but pressure castings have also been used successfully in this type of construction.

Surface textures and engraving strongly favor pressure casting. Many effects that require expensive after-operations in other processes are obtained in pressure casting for a modest extra cost of detailing in the original hob.

A solid blown part will require sliding pinch-off at the blow point to seal off the finished part. Needles may be introduced practically anywhere on a part. Additional problems arise, however, if, for instance, the needle is desired in an area adjacent to water lines or cooling area or at the top or bottom of the part requiring movement of the needle during the cycle.

The type of machine available has a strong influence upon the blow opening used. Some machines, for instance, use needle blowing exclusively, machining off the excess material to provide larger openings when required.

Single plane, vertical parting lines are the norm for most blow molds. In some cases the product is blown at an angle other than its normal mid-axis. This requires tipping of the cavities and can be accomplished by all methods of construction.

In some cases, as with a figurine, it is necessary to have a parting line that is not in a single plane. Such cavities would be best cast in steel or beryllium copper. The casting will give a close miter economically and the steel or beryllium copper will give strength to the parting line to resist the wear and tear this type of die is subjected to in operation.

A close capacity specification also seriously influences die construction. Where multiple molds are required, capacity from one die to another must be closely held. In general, some of the casting processes have been more successful in reducing this factor than duplicating or hobbing. For final capacity adjustment an inserted bottom plate, or other adjustable insert, is advisable. By this method slight changes can be made on each die insert without noticeable variation in finished parts.

An interchangeable fit with other parts requires serious consideration in die construction. There are plug fits, screw and snap-type caps, snap beads, doll parts, etc. It is strongly advisable to use inserts for the interchangeable portion. This would allow for changes of inserts to accommodate different shrinkage conditions, different materials, etc.

Physical characteristics of the finished part may influence die construction. A requirement for high resistance to stress cracking, for example, may require variable temperature control in different parts of the die such as the bottom plate or neck inserts. Such a construction would appear more economical using an inserted die rather than a solid casting.

Parts requiring detailed printing are usually desired with a high surface luster. Luster can be produced by sand-blasting or other roughening of the surface. The roughening of the surface permits the release of trapped air. A die with several inserts will often accomplish the air release without roughening. In severe cases, such as deep engraving, etc., vent holes of .005" to .010" are necessary. Confliction of vent holes with cooling channels is a common obstacle. Teflon, plastic resin and various other coatings have been successfully used on dies to accomplish luster. Plating has been used but does not seem to be the answer as it is in injection molding. Cast iron dies, however, definitely should be plated for both release and surface luster. Silicon release agents will cause peeling if the product is to be printed or painted.

The Equipment—How It Affects Mold Costs

The available type of equipment on which a given job can be run has a profound influence on the construction of dies. Some machines use only a single cavity; others require a minimum of 8 to 12 cavities regardless of the size of the item. For small items, multiple cavities as high as 16 have been used, and larger numbers can be accomplished.

Obviously, large numbers of cavities are feasible only when production quantities are high. More cavities mean more total investment, but the difference in die cost is often more than offset by a reduction in piece price on a multiple cavity setup.

A difference of $2,000 in die costs would be more than offset by a $1.00 per/M price differential on a 2½ million lot.

At $10.00 per hour machine time a small item would accomplish this by an increase from 1000 per hour to 1250 per hour. Such differences in production speeds are quite common from one type of setup to another.

The position of the blowing opening has some bearing on die construction. Many machines can accomplish top, bottom or side needle blowing. There are, however, some machines wherein one or two of these cannot be accomplished without major changes. For example, some machines will blow only from the bottom, others only from the top—others use needle blowing exclusively.

edited by **Ernest J. Csaszar,** *Newark Die Company*

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Reinforced Plastics

Effects of Stress Concentration on the Strength of Reinforced Plastic Laminates

ERIC L. STRAUSS
THE MARTIN CO.

For the engineer: Practical design information to aid in the selection of safe stress levels when working with reinforced plastics

One of the important factors to be considered in designing a reinforced plastic structure is the notch sensitivity of the material. Fatigue cracks in metals tend to originate at points of stress concentrations, such as flaws in the material, surface blemishes, corner fillets or holes. In reinforced plastics, stress concentrations induce premature failures not only after numerous load cycles but also during application of a steady load.

Since stress concentrations cannot be avoided entirely in a structural plastic resin, the working stresses must be sufficiently low to preclude failures in the vicinity of stress concentrations. However, little information is available to guide a designer in the selection of safe stress levels. An experimental program was undertaken to determine stress concentration effects in reinforced plastic laminates. The material selected for this study was a ¼-in. thick plastic laminate containing 12 layers of 181 glass fabric and a polyester resin. The program was designed to facilitate a comparison of the tensile, compression, and flexural strengths of specimens containing various stress concentrations with the strengths of control specimens of standard shapes. Failing stresses were calculated, based on the net cross-sectional area of the specimens. Results presented in this article represent the averages of three specimens. **By adhering to normal shop practices in the fabrication of the test pieces, the resulting test values became meaningful as practical design data.**

TABLE 1

Tensile Strengths of Glass Fabric-Polyester Laminates Containing Holes of Various Diameters

Hole Diameter (in.)	Hole Diameter / Spec. Width 0.050	0.083	0.143	0.250	0.400	0.571
1/16	36,000	38,700	38,500	—	—	—
1/8	33,500	35,700	37,700	35,400	—	—
1/4	—	29,000	30,600	30,100	35,900	—
1/2	—	—	27,100	30,000	30,000	—
1	—	—	—	21,700	—	30,800

Nominal Tensile Strength = 40,700 psi

TABLE 2

Strengths of Glass Fabric-Polyester Laminates Containing Holes of Various Diameters

Hole Diameter (in.)	Hole Diameter / Spec. Width 0.050	0.078	0.083	0.143	0.250	0.400
				Flexure—Nominal Strength = 65,200 psi		
1/16	57,700	58,100	—	—	—	—
1/8	56,700	—	57,500	56,000	—	—
1/4	53,700	—	57,200	54,600	56,600	—
1/2	—	—	47,500	53,700	—	51,700
				Compression—Nominal Strength = 30,300 psi		
1/16	24,600	45,400	—	—	—	—
1/8	—	—	15,500	39,100	—	—
1/4	—	—	—	32,200	28,600	—
1/2	—	—	—	—	—	26,400

Drilled Holes

The isolated holes ranged from 1/16 in. to 1 in. in diameter. The tensile test data presented in Table 1 exhibits a pronounced decrease in specimen strength with increasing hole size. The normal tensile strength of the laminate was 40,700 psi. Strengths of specimens containing a 1/16-in. diameter hole ranged from 36,000 to 38,700 psi. A 1-in.-diameter hole reduced the tensile strength to a value as low as 21,700 psi. Figure 1 illustrates that the strength drop-off with increasing hole diameter holds for flexure and compression as well as for tension. However, the flexural strength diminishes more gradually than either the tensile or compression strength.

Specimen Width

A series of specimens was prepared in which both specimen width and hole diameter varied such that the net width (specimen width less hole diameter) remained constant. Results of tension and flexural tests of this specimen group are compared with the normal strengths in Fig. 2. The normal values are represented as 100%. The decrease in strength with increasing hole diameter is again noted. A ¾-in.-diameter hole caused a 21% loss in the flexural strength and a 26.5% loss in the tensile strength of the laminate.

Hole Diameter to Specimen Width Ratio

The ratio of hole diameter to specimen width (d/w ratio) was an additional parameter in the stress concentration investigation. Hole diameters ranged from 5 to 58% of specimen width. The tabulated tensile test results in Table 1 show a general increase in failing stress with increasing d/w ratio. Flexural and compression test results, summarized in Table 2 display the same general trends as the tensile test results.

Hole Location

The effects of hole location were investigated with another group of

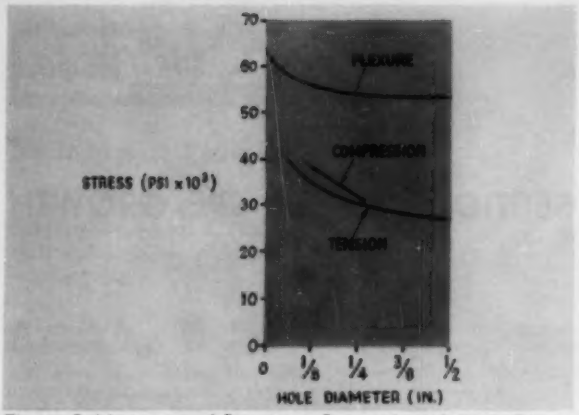


Figure 1. Variation of Specimen Strength with Hole Diameter (Hole Dia/Specimen Width = 0.143).

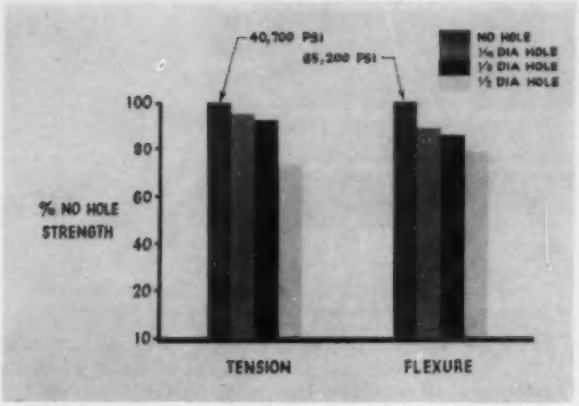


Figure 2. Strength of Polyester Laminates as a Function of Hole Diameter (Constant Net Width = 0.750 in.).

test specimens. The position of a ¼-in. diameter hole in a 3-in. wide specimen was varied from a location at the specimen center to a location 5/16 in. from the specimen edge. The data, plotted in Fig. 3, indicate no significant strength difference between on-center and off-center locations of holes.

Holes in a Single Row

Specimens with multiple holes in line were tested to ascertain whether a row of holes behaves in a similar manner as isolated holes.

First tested were two configurations, having identical d/w ratios of 0.125:

	Failing Stress (psi)
¼-in.-dia holes, 1-in. spacing	29,500
¼-in.-dia holes, 2-in. spacing	26,200

The two specimens with d/w ratios of 0.250:

	Failing Stress (psi)
¼-in.-dia holes, 1-in. spacing	29,300
½-in.-dia holes, 2-in. spacing	25,700

In each group, strength decreases with increasing hole diameter, paralleling the behavior of single holes.

Next specimens of identical 4-in. width having ¼-in.-diameter holes were compared:

	Failing Stress (psi)
2-in. spacing, d/w = 0.125	29,300
1-in. spacing, d/w = 0.250	25,700

Here, as with single holes, the strength increases with increasing d/w ratio.

Finally, we directly compared multiple holes with equivalent single-hole specimens:

	Failing Stress (psi)
(1) ¼-in.-dia holes, 1-in. spacing	29,300
¼-in.dia hole, 1-in. specimen width	30,100
(2) ½-in.-dia holes, 2-in. spacing	25,700
½-in.-dia hole, 2-in. specimen width	30,000

In both instances, the single-hole specimen failed at a higher stress level than the comparative multiple-hole specimen. It is reasoned that the larger number of holes in the multiple-hole specimens increases the probability of early crack formation and propagation.

TABLE 3
Variation of Specimen Strength with Notch Radius

3/4-in. Constant Net Width

Notch Radius (in.)	Tensile Strength (psi)	Compressive Strength (psi)
1/16	37,500	38,500
1/8	35,700	35,100
1/4	32,200	30,400

1/8-in. Notch Radius

d/w Ratio*	Tensile Strength (psi)	Compressive Strength (psi)
0.083	24,200	—
0.143	28,400	34,200
0.250	35,700	35,100
0.333	35,300	—

* d = 2 x Notch Radius

(*Continued on page 898*)

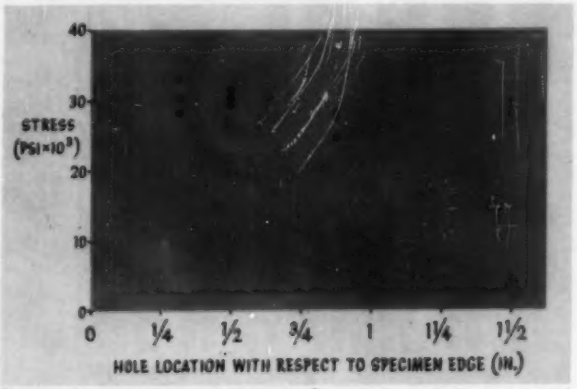


Figure 3. Variation of Tensile Strength with Location of 1/4-in.-Diameter Hole.

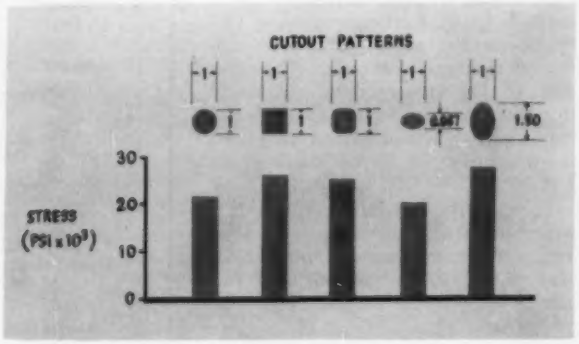


Figure 4. Tensile Strength of Polyester Laminates as a Function of Hole Cutout Pattern (Constant Net Width = 3 in.).

National Action

GEORGE W. MARTIN, SPE NATIONAL FIRST VICE-PRESIDENT, AND ADMINISTRATOR, NEW SECTION DEVELOPMENT COMMITTEE.

NEW SECTIONS SPARK SPE GROWTH

SPE's By-Law B-55 states that "The New Section Development Committee shall encourage and assist in the formation of Sections in areas where need can be demonstrated".

This hard-working—although often unheralded—National Committee is a primary force contributing to the sound growth of our Society. Although our Membership Committee performs an outstanding job in the solicitation of new members, the problem of keeping these new members actively on the SPE Roster is an ever-present one. To carry out the objectives of our Society it is essential that members have an opportunity to attend regular Section meetings held within convenient distance of their homes.

Far too often our members are faced with the necessity of traveling great distances to attend the nearest Section meeting. The result is frequently discouragement, and sometimes, resignation. By the same token, of course, the Membership Committee is hampered severely in such areas due to the lack of nearby Sections to spread the gospel. The solution to these problems, by fostering and developing new Sections is the primary objective of the New Section Development Committee.

In the past, the Society has indeed been fortunate in having men of the caliber of Joe Healey, Bill Dunnican and, most recently, Ross Dean to lead the many, many other members who have worked so diligently on this very important task. Certainly much of the credit for the rapid growth of our Society belongs to these men. In the case of Joe Healey who performed this task for many years, Council has made him a "Distinguished Member" in recognition of his outstanding effort.

Ross Dean certainly performed a superb job, directly resulting in the addition of our newest Florida Section, the Tri-State Section and the Bartlesville-Tulsa Section. He was also instrumental in the successful splitting off of the Akron and Cleveland Sections. In addition, we expect Sections in many other areas to materialize soon. Monterrey, Mexico, is now a Section and there is promising activity in Western Michigan, in Arizona, in West Virginia and in Southwestern Canada.

While extending credit for these fine achievements, we cannot overlook our National Office staff in the outstanding co-operation they contribute to this Committee.

And finally, full credit is due to individual members and non-members who have devoted so unselfishly of their time and effort to the development of Sections in their own areas.

If the Society of Plastics Engineers is to represent every plastics engineer in the Country, we must continue this work and, in so doing, earnestly solicit the aid of all SPE members.

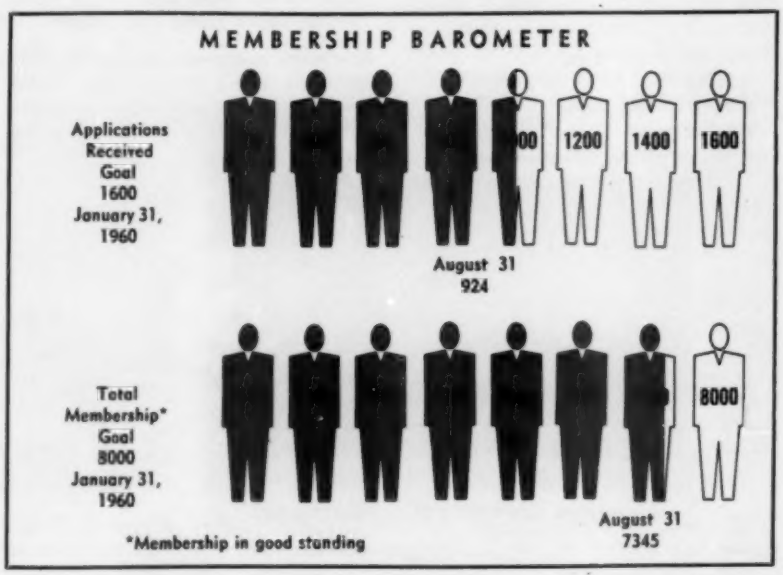
Our newly formed Public Relations Committee will provide powerful assistance by spreading the Society's doctrine throughout vast untouched areas of our Country and, for that matter, of the world. Once interest has been aroused in an area it becomes the Committee's responsibility to move in and do the development work required.

Our new Section Development Procedures Manual—originally prepared by our Distinguished Member, Joe Healey is now in the process of being revised and reprinted. It is our intention that every Section Membership Chairman will receive a copy of this manual to enable him to assist our new Section Development Committee whenever called upon to do so.

Naturally, the Committee leans very heavily on the Sections adjacent to the area being considered for a new Section. Potential new members look toward these Sections for guidance and example.

Our new Committee Chairman, since July 1st, is Col. Oscar B. Yorker, National Councilman, representing our Rocky Mountain Section. He is very capable of carrying on this important work.

Chairman Yorker will appreciate guidance or suggestions from any member or non-member that might lead to the development of a new SPE Section. Please write directly to Col. Oscar B. Yorker, c/o Yorker & Sons, Inc., 601 East 48th Avenue, Denver 16, Colorado.



edited by **Thomas A. Bissell**
SPE Executive Secretary

RETEC

Stability of Plastics

Sponsored by the Baltimore-Washington Section
Tuesday, December 1, 1959
National Academy of Sciences Building
Washington, D. C.

The Baltimore-Washington Section, SPE, in conjunction with the Prevention of Deterioration Center of the National Academy of Sciences, has scheduled a Regional Technical Conference entitled "Stability of Plastics". The one-day meeting will be held Tuesday, December 1, 1959 in the auditorium of the National Academy of Sciences Building, 2101 Constitution Avenue, Washington, D. C. Dr. Herman Mark, Director, Polymer Research Institute, Polytechnic Institute of Brookyln, will preside as moderator of the program.

RETEC objective: To present a program covering current concepts of mechanisms governing natural and synthetic polymer stability and degradation. Degradation under the conditions of mechanical processing, ultraviolet or other radiational exposure, elevated temperatures, and enzymatic attack will be among the topics covered by well-known contributors to these fields.

TENTATIVE PROGRAM

9:00 AM	Welcome Mr. Myron DeFries, Baltimore-Washington Section RETEC Chairman
9:05 AM	Work of the Prevention of Deterioration Center Dr. Carl Wessel, National Academy of Sciences
9:20 AM	Introduction to RETEC Dr. Herman Mark, presiding
9:30 AM	Technical Papers
12:00-2 PM	Luncheon—Arlington Towers, Terrace Room Mr. George Flanagan, President, Baltimore-Washington Section, introducing National Officers of SPE: Messers. Fred Sutro, Jules Lindau and Tom Bissell
2-4:30 PM	Technical Papers

In addition to the abstracts appearing on this page, a paper by Dr. Leo Wall, National Bureau of Standards, on "Thermodynamic Aspects of Polymer Degradation" will be presented. Other abstracts will appear in the November issue of *SPE Journal*.

Arrangements for accommodations at the Washington and Willard Hotels (within walking distance of the meeting) and at the Windsor Park Hotel and the Arva Motel have been made for those desiring them. Registration fee is $10.00 ($7.50 for government employees) including luncheon, transportation to and from the luncheon, and a copy of a reprint book containing comprehensive summaries of papers presented at the meeting.

All SPE members in Sections of the eastern region of the United States and Canada (Mississippi Valley or east) will receive letters of invitation and advance registration forms. Others desiring information, may contact: Mr. Myron DeFries, RETEC Chairman, Atlantic Research Corporation, Alexandria, Virginia.

Advance registration and hotel accommodations may be obtained through: Mr. Stanley Prosen, Naval Ordnance Laboratory, White Oak, Silver Spring, Maryland.

Technical Conferences

Mechanism of Ultraviolet Stabilization of Plastics

J. H. CHAUDET, G. C. NEWLAND, H. W. PATTON, and J. W. TAMBLYN, RESEARCH LABORATORIES, TENNESSEE EASTMAN COMPANY

Most ultraviolet stabilizers for plastics appear to act as inert, inner filters. Some of the ways by which these stabilizing molecules can absorb ultraviolet energy and release it without harming the surrounding polymer molecules have been considered. The following types of stabilizer were studied:

(a) Fluorescent molecules
(b) Molecules capable of undergoing energy-absorbing rearrangements
(c) Molecules containing chelate rings
(d) Stable free radicals

A study has been made, by nuclear magnetic resonance spectroscopy, of a number of derivatives of 2-hydroxybenzophenone. The positions of the hydroxy proton resonances for the more effective light-stabilizers fell at lower field values

Polymer Stability—Its Effect on Processing and Properties

H. S. KAUFMAN AND E. C. SCHULE, ALLIED CHEMICAL CORPORATION, CENTRAL RESEARCH LABORATORY

In the course of fabrication and processing, high polymers may be subjected to thermal and mechanical treatment which result in molecular weight degradation and morphological change. The effects of this phenomenon as related to injection molding and extrusion will be discussed in terms of polymer properties, including crystallization and orientation. Examples will be drawn from experience with polyamides, polyethylenes and polychlorotrifluorethylene.

Degradation of Natural Polymers

E. T. REESE AND MARY MANDELS, QUARTERMASTER CORPS., U. S. ARMY

Degradation of natural polymers is usually the work of biological agents, and in this respect it differs from the degradation of most synthetic polymers. The degradation is a depolymerization resulting from an enzymatic hydrolysis, the final products being water soluble and which are capable of serving as food for the degrading organism.

Many of the natural polymers used in textiles have a highly oriented, or crystalline, structure. As a result, there is a natural resistance to water absorption. The tight packing of chains also prevents the ready penetration of the large catalyst molecule. Any change in the fiber which leads to a loosening-up of the structure permits greater water uptake and increased diffusion of the enzyme, and so increases its susceptibility to degradation.

The Weathering of an Elastomeric Polyurethane

C. S. SCHOLLENBERGER AND K. DINBERGS, B. F. GOODRICH RESEARCH CENTER

Estane VC, a strong, tough, thermoplastic, "virtually crosslinked" polyurethane elastomer shows a certain degree of susceptibility to the weathering process as do other unprotected linear polymers. Using the (dry) Weatherometer as a substitute for sunlight, and suitable optical filters, it was possible to demonstrate that the incident solar rays deleterious to Estane VC are of the wavelength range 340 to 410 m (critical region) a region in which the polymer actually shows little absorption. The effectiveness of custom-synthesized compatible compounds which absorb strongly in the critical region as weather resisters in Estane VC is described.

(*additional abstract on page 902*)

Plastics Around the World

This month's column has been made possible through the contributions of these abstracters:

Alfred L. Alk (Materie Plastiche, Seleplastic)
Charles Imig (Kunststoffe)
Alfred Rexer (Plastics)

Germany

KUNSTSTOFFE

MARCH 1959

Basic Laws for Plastics Screw Extruders, Part III—Dr.-Ing. Gerhard Schenkel

This is the final paper of a series of three papers dealing with this subject. Mathematical treatment is continued to show that each of the three extruder zones behave differently but at the same time must be considered in relationship to one another. Examples of how this theory applies to different thermoplastic materials is presented.

APRIL 1959

Rheological Construction of Simple Extrusion Dies, Part I—Dr.-Ing. Gerhard Schenkel

Two requirements must be met in the construction of common extrusion dies. First, the plastic melt must be diverted from the axial direction on the extruder screw to some direction without changing the characteristics of the melt. Second, an internal mandrel must be placed in the stream to form a cylindrical shape. Discussed, among other topics, is pressure equalization with various inserts, different cross-head designs, and different mandrel designs. The second part of this paper will describe ways of removing marks caused by mandrel supports.

Great Britain

PLASTICS

MARCH 1959

Heatsealing and High Frequency Welding of Plastics—H. P. Zade

The second part of a continuing article covers the various types of heatsealing equipment.

Italy

SELEPLASTIC

MARCH 1959

ICI and the Conveyor Belt for Russia—

ICI announces the use of its Terylene polyester fiber in a huge conveyor belt over six miles long and up to six feet wide which is being fabricated in England for use in a Russian iron mining area. The belt consists of 8 to 14 layers of rubber covered Terylene. The largest section can withstand a maximum working tension of 66 tons and can transmit more than 1000 HP.

MATERIE PLASTICHE

APRIL 1959

Technological Properties of Moplen—F. Ranalli, G. Crespi

Moplen can be processed in common plastic fabricating equipment after the optimum operating conditions for the particular machine have been determined. At elevated temperatures polypropylene is subject to degradation and oxidation which makes the finished article fragile. Attention to maximum operating temperatures and the addition of anti-oxidants alleviates both problems. Injection moldability of Moplen is compared with polystyrene and polyethylene under various conditions. Test methods are described. Not only is the melt index of the polypropylene a factor in moldability, but also its degree of isotacticity. Operating conditions and injection cycles lie generally between those required for high impact styrene and low density polyethylene. The piece weight should be kept at a ratio of 3:1 to 10:1 of the cylinder capacity. Generally Moplen works better at higher injection pressures and high flow velocity. Temperatures from 200° to 270°C are required with 270° being the maximum practical because of breakdown. Plasticizing chambers need not be redesigned, although certain hints are given for increasing effectiveness of the molds. Moplen need not be predried. Molded articles shrink 1.1 to 2%. Moplen can be extruded without difficulty at temperatures between 190° and 270°C in long barrel extruders with 3 or 4:1 compression. Pipe can be produced in equipment normally employed for polyethylene or PVC. Moplen can also be used to produce blown and flat films, and for thick sheeting suitable for vacuum forming, for wire insulation, as anti-corrosion lining for metal containers

(*Continued from page 895*)

Comparison of Drilled, Countersunk, and Riveted Holes

Countersunk holes were machined to standard tolerances for 100° flush head rivets. Countersunk and riveted specimens were prepared in the following manner:

(1) 2024 aluminum alloy rivets were dipped in a room temperature curing epoxy adhesive and inserted into the drilled and countersunk holes.

(2) An aluminum washer was placed over the rivet shank and the driven head was formed. A mean hole diameter was utilized in calculating failing stresses of specimens containing countersunk holes.

Countersinking reduced the failing stress approximately 5% in both tension and compression when compared with drilled holes. Flexural test results were inconclusive. Insertion of a rivet resulted in a significant increase in specimen strength for all three loading conditions. The greater strength was expected in compression and flexure since the rivet is capable of transmitting a compressive load across the hole. In tension, the adhesive apparently bonded the rivet to the sides of the hole so that a tensile force could also be transmitted by the rivet across the hole.

Spacing between Rows (Back Pitch)

A series of specimens containing multiple rows of holes was tested in tension to determine the effects of varying spacing between rows (back pitch). Specimens were 4 in. wide, containing three rows of staggered holes with 1-in. spacing between holes. Tensile test results are summarized below and compared to a specimen of similar configuration containing a single row of holes.

	Failing Stress (psi)
¼-in.-dia holes, 1-in. between rows	24,200
¼-in.-dia holes, ½-in. between rows	26,900
¼-in.-dia holes, ¾-in. between rows	25,400
¼-in.-dia holes, single row of holes,	29,300

(*Continued on page 900*)

RETEC

Technical Conferences

Plastics in Packaging

Sponsored by The Golden Gate Section
Thursday, November 19, 1959
Claremont Hotel, Berkeley, California

PURPOSE

Two vital industries will combine to present a one-day conference on the West Coast November 19. Called "Plastics in Packaging," the technical meeting will consist of reports by top-flight experts in both plastics and packaging. Sponsored by the Golden Gate (San Francisco) Section of SPE, the meeting will be held at the Claremont Hotel in Berkeley, California.

Headlining the program will be McKay McKinnon, Jr., Chief of District for the Food and Drug Administration, with headquarters in San Francisco.

Speakers at the mid-day luncheon will be Fred C. Sutro, Jr., Spencer Chemical Co., National President of SPE, and John Delmonte, Furane Plastics, Inc., Los Angeles, Calif., Adminstrator of Meetings, SPE. Mr. Sutro is also co-author of one of the technical papers on the program.

Morning Session (Registration 8:00 A.M.)

9:00 AM—INTRODUCTION

ROGER JENNINGS, MODERATOR
Polytron Corporation, Richmond, Calif.

9:15—MILITARY PACKAGING,

J. B. Smith, Jayro Corporation, San Francisco

New developments of reinforced plastic military packaging, including a discussion of current developments using urethane foams as an integral part of the case and other cushioning materials. An exploration of the potential of technical plastic applications for electronic and instrumentation packaging.

9:45—LOW MOLECULAR WEIGHT POLYETHYLENE FOR PAPERBOARD COATINGS,

R. Rosenbaum, Semet-Solvay Petrochemical Div., Allied Chemical Corp., New York

Polyethylene and paper combinations are well established in the packaging field and combinations of polyethylene and paperboard are now being explored. The low molecular weight polyethylenes alone or in combination with waxes and resins have found paperboard coating applications because of good functional properties combined with ease of applications on conventional equipment. Abrasion resistance, hardness, blocking, gloss, gloss retention, tensile strength and chemical resistance of wax is improved by the addition of polyethylene.

10:15—NEW DEVELOPMENTS IN RIGID PLASTIC CONTAINERS,

R. J. Lee and L. E. Tallman, The Dow Chemical Company, Midland, Michigan and Torrance, Calif.

At present there are several areas in packaging where rigid or semi-rigid plastic containers are starting to make extensive inroads. Some of these would include linear polyethylene blown bottles for household products, foamed polystyrene for hot beverage cups, formed high impact containers for dairy and delicatessen products, formed components for composite cans and molded containers for hermetic packaging of selected products. Some of the economic factors and technical problems in material selection and product protection will be discussed.

10:45—Break

11:00—AUTOMATIC OVERWRAPPING WITH THERMOPLASTIC FILMS,

C. B. Tanc, Union Carbide Plastics Co., Los Angeles, Calif.

Automatic packaging with thermoplastic films may present the largest presently known single potential market for plastic packaging material, including such films as polyethylene, polypropylene and polyvinyl chloride. Use of automatic equipment handling plastic films, has already begun in the field of bread wrapping, frozen foods, meats, baked goods, stationery, soft goods and produce. The size of the market is enormous, with some estimating the possible immediate potential for bread wrap alone to be as high as 60 million pounds of film annually.

11:30—THE TOXICOLOGY OF PLASTIC PACKAGES AND FOOD ADDITIVES LAW,

Dr. Louis C. Barail, consultant, New York, N. Y.

An outline of the food additives law and its requirements explains in detail how it involves plastic packages used by the food, drug, chemical and allied industries; description of the toxicological methods approved by the Food and Drug Administration, how their results are interpreted and how their early application can save manufacturers and users of plastic wrappers and containers many headaches, troubles and financial losses.

12:00-2:00 PM—Luncheon

Speakers—Fred C. Sutro, Spencer Chemical Co., Kansas City, Mo., National President, SPE; John Delmonte, Furane Plastics, Inc., Los Angeles, Calif., Administrator of Meetings, SPE

2:00—VIEWS OF THE FOOD AND DRUG ADMINISTRATION,

McKay McKinnon, Jr., Chief of District, Food and Drug Administration, San Francisco, Calif.

(Abstract not available. Because of rapid-fire developments in this field, Mr. McKinnon's presentation calls for last minute preparation.)

3:00—CHILL ROLL TECHNIQUES OF PRODUCING POLYETHYLENE FILM,

Fred C. Sutro, Jr., and D. L. Duncan, Spencer Chemical Co., Kansas City, Mo.

During the past year, there has been tremendous interest in polyethylene film produced by the chill roll extrusion method. Many of the questions have centered around the marketing problems connected with a film which has some definite differences from film produced by the more conventional blown tubing method. The two primary differences are in the areas of optical properties and strength properties. Certain surface differences are also noted, but these are relatively minor from a selling standpoint. Chill roll more nearly resembles cellophane than any other type of polyethylene, particularly when the film is made from medium density resins. The characteristics of these films are such as to allow the use of polyethylene film as a genuine overwrap material for the first time.

3:30—Break

Conference separates into two concurrent sessions

SECTION A

ROGER JENNINGS, MODERATOR
Polytron Corp.

3:45—DESIGNING THE PACKAGE,

Cornelius Sampson, Cornelius Sampson and Associates, San Francisco

Self-service and supermarkets have created a pre-packaged world of merchandising. Plastics offer opportunity to make full use of the natural eye and color appeal inherent in the product. Product and weight. Brand identification. Product name and description. Supplemental information. Legal information. Research competition and determine positive sales factors of product. Slide examples of good and poor design treatments with case histories of meat, produce, and fruits use of plastic wraps or containers.

4:15—HEAT-SHRINKABLE AND POLYMER-COATED POLYESTER FILMS,

Paul G. Stephan, E. I. du Pont de Nemours & Co., Inc., Wilmington, Del.

A heat-shrinkable polyester film combines the properties of the standard form of the film with extremely close conformation to the product package. Physical and chemical properties of the film are discussed and related to specific packaging requirements. Alternate methods of applying the heat required in the shrinking process are described. Potential uses for the film are analyzed together with application experience. A description of certain other types of polyester film—modified from the standard for adaptation to specific uses—is included.

(Continued on page 903)

Walter A. Gammell, Sr.,

Chairman, Plastics in Electrical Insulation PAG

Participation Invited in Electrical Insulation PAG Task Groups

What can you as an individual do for SPE and what can SPE do for you? As you know, SPE has been divided into various fields of interest —our particular field is embedment and encapsulation for electrical insulation. This field in turn has sub groups which cover specific phases of interest.

To help you become better acquainted with the work in progress in the Professional Activity Group on Plastics in Electrical Insulation, the task groups with their chairmen are listed below:

RESINS—
Dr. William J. Belanger
Jones-Dabney Company
1481 South 11th Street
Louisville, Kentucky

FILLERS—
Dr. R. G. Black
Westinghouse Electric Corporation
Materials Engineering—K-70
Insulation Development
East Pittsburgh, Pennsylvania

PROCESSING METHODS—
Mr. August L. Kraft
Automatic Process Control
1170 Morris Avenue
Union, New Jersey

MOLDING AND COATING (FLUIDIZED BED) METHODS—
Mr. George F. Chadwick
Speer Carbon Company
Research Laboratory
Packard Rd. & 47th St.
Niagara Falls, N. Y.

The object of a task group is to carry out cooperative projects which will be of benefit to any member of SPE. The first three groups have been active for several months and each group has a small corps of workers. The fourth group under George Chadwick is just getting launched and is looking for active workers.

There are many projects being considered. The one being carried on at the moment is a bibliography project. The members of each task group, under the leadership of the above-listed chairmen, are preparing an index of the articles available in their respective areas. Much of this project will be completed during 1959, and the completed bibliographies will be published in the SPE Journal and made available in printed form to any SPE personnel having an interest in them. Each of the task groups has openings for additional membership and, therefore, invite you to join them. Further details of the activities and membership of the individual task groups are available by contacting the task group chairman.

It is hoped that many of you will contact these chairmen and become active in our Professional Activity Task Groups. We know that it will be valuable to you both technically and otherwise, since you will have a better opportunity to participate in future conferences such as our Fort Wayne Conference and in some of the other task group projects.

All who attended the May RETEC in Fort Wayne know of the fine turnout and of the excellent reception the various papers received. As this type meeting tends to bring people back for the next one, we hope that you are planning to attend the ANTEC in Chicago in January, 1960.

To those who did not get to the Fort Wayne meeting, we would like to state that as meetings go, this one was one of the better ones in recent years—the subject matter was particularly timely, concisely presented and well received. The whole affair was well organized by the local members.

Come to Chicago and see for yourself what these people are doing for you and find out where you can assist. In the meantime, we do suggest that you write the above chairmen of the task groups and get involved with a project.

Electrical Insulation Conference

The 2nd National Conference on the Application of Electrical Insulation will be held at the Shoreham Hotel, Washington, D. C., December 8-10, 1959.

(*Continued from page 898*)

These test data reveal no appreciable difference in the strength of multiple-row specimens containing spacings of 1 in., ½ in. and ¼ in. between rows. However, the single row specimen was considerably stronger than any of the triple row specimens. This confirms the previous conclusion that for specimens containing equal hole size and hole spacing, the one having the largest number of holes will fail at the lowest stress level.

Cutout Shape

To facilitate a direct comparison of the influence of cutout shape, all specimens were 4 in. wide and contained cutouts of 1-in. width. Circular, rectangular and elliptical cutouts were investigated. Tensile test results are illustrated in Fig. 4.

The elliptical cutout whose major axis ran perpendicular to the loading direction caused the greatest reduction in tensile strength while the ellipse whose major axis ran parallel to the loading direction exhibited the highest tensile strength. The strengths of specimens containing rectangular cutouts with either square or rounded corners were approximately equal and were appreciably higher than the strength of the specimen with the circular cutout.

Notch Radius

Specimens with semicircular notches were in many ways similar to specimens containing holes of equal radii. Test results are summarized in Table 3. In the series of specimens of constant ¾-in. net width, strength decreased with increasing notch radius. When notch radius was held constant, strength increased with increasing d/w ratio where d = 2 x notch radius.

Fillet Radius

A standard tensile specimen for plastic laminates has a ½-in.-wide gage section which is 2-¼ in. long and flares out at both ends to a ¾-in. width. The transition is formed by arcs of 3-in. radius. Effects of variations in fillet radius were investigated by essentially retaining the shape of the standard tensile specimen but varying the transition radius from 10 in. to ¼ in. Test results show that strength remains unaffected by fillet radii ranging from 10 in. to 1 in., but decreases when fillet radii become smaller than 1 in. The standard tensile specimen containing the 3 in. fillet radius yielded strength values of 40,700 psi. A specimen containing a ¼-in.-diameter fillet radius failed at 36,000 psi, representing a 10% loss in strength.

Edited by George Lubin
Grumman Aircraft Co.

Technical Meetings Calendar

16TH ANTEC

ANNUAL TECHNICAL CONFERENCE—January 12-13, 1960, The Conrad Hilton, Chicago, Ill. Sponsored by the Chicago Section. For more information, contact General Chairman Franklin L. Fine, Rohm & Haas Co., 5750 W. Jarvis Ave., Chicago 31, Ill. Request for papers on page 275 of the April SPE Journal.

1959 NATEC

NATIONAL TECHNICAL CONFERENCE—October 13-14, 1959. The Ambassador, Los Angeles, Calif. For more information, contact General Chairman, Jack C. Fuller, c/o Chemtrol Co. 10872 Stanford Ave., Lynwood, Calif. See program and abstracts page 815, September, 1959 SPE Journal.

RETECS

VINYLS—October 7, 1959, Cleveland Engineering Society, 3100 Chester Ave., Cleveland, Ohio. Partial program page 745, August SPE Journal. For further information contact Jack R. Pecktal, 13212 Shaker Square, Cleveland 20, Ohio.

PLASTICS FINISHING—October 16, 1959, The Niagara Hotel, Niagara Falls, N. Y. Sponsored by the Buffalo Section in cooperation with the Plastics Finishing PAG. For program, see page 823, this issue. Chairman Gordon K. Storin, 3 Forest Rd., Lewiston Heights, Lewiston, N. Y.

PLASTICS IN THE SHOE INDUSTRY—November 4, 1959, Statler Hotel, St. Louis, Mo. Sponsored by the St. Louis Section. For program information, see page 820, this issue. Otto Wulfert, Chairman, c/o Wagner Electric Co., 6400 Plymouth Ave., St. Louis 14, Mo.

PLASTICS IN PACKAGING—November 19, 1959, Berkeley, Calif. Sponsored by the Golden Gate Section. For program, see page 899 this issue. Chairman, Frank D. Allen, L. H. Butcher Co., 15th & Vermont Sts., San Francisco 1, Calif.

STABILITY OF PLASTICS—December 1, 1959, National Academy of Science Bldg., 2101 Constitution Ave., NW., Washington, D. C. Sponsored by the Baltimore-Washington Section. For information write to Chairman Myron G. DeFries, Atlantic Research Corp., Alexandria, Va.

—EXTRUSION WORKSHOP—

CENTRAL OHIO—Third Extrusion Workshop sponsored in cooperation with the Extrusion PAG.

Date: October 20, 21, 1959

Place: Ohio State University, Ohio Union Conference Theater

Fee: $35.00 to include registration, 2 lunches, dinner and coffee.

Speakers: E. Veazey, Dow Chemical, E. Bernhardt and H. Kessler, The Du Pont Co., B. Maddock, Union Carbide Plastics Co., F. Nissel, Prodex Corp., M. Underwood, Monsanto Chemical Co., and R. Sackett, Hartig Extruders. (Moderator)

Attendance: Anyone from any Section may attend.

Registration: Send fee, name, address and company affiliation to Chairman Mr. Robert O. Zimmerman, Plastex Co., 3232 Cleveland Ave., Columbus, Ohio. Deadline is September 30, 1959

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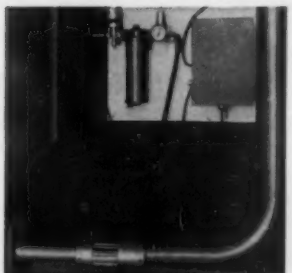

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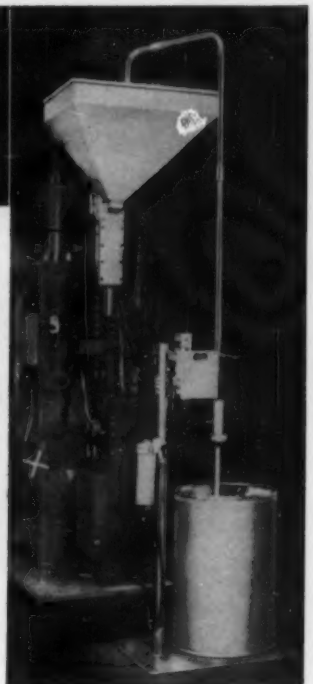

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(abstracts continued from page 897)

Ultraviolet Degradation of Plastics and the Use of Protective Ultraviolet Absorbers

R. C. HIRT, N. D. SEARLE AND R. G. SCHMITT, CENTRAL RESEARCH DIVISION, AMERICAN CYANAMID COMPANY

The stability of plastics to ultraviolet radiation and the use of protective ultraviolet absorbers to improve this stability necessitates a study along several lines. These include the ultraviolet spectral energy distribution of the source, the degradation as a function of the wavelength of incident radiation, and the measure of the effectiveness of the protective ultraviolet absorbers.

Photochemical decomposition of the protective ultraviolet absorbers has been studied under various sources with severe irradiation intensities. The decomposition reaction appears in some cases to be dependent mainly on the energy absorbed and in other cases to approach true first-order kinetics.

Welcome Monterrey

It is now the Monterrey Section, without the provisional, that is. Our newest section south of the border has completed full requirements for Section status. Two regular meetings have been held (see Section News, page 906) and the new Section would like to hear from speakers who can address the group in Spanish on subjects relating to the plastics industry. Please address communications to: Ing. John P. Brecker, Ecuador #612–Colonia Vista Hermosa, Monterrey, N.L. Mexico

VINYL and FINISHING RETEC BOOKS JOIN SPE PUBLICATIONS

Announcing

Vinyl Plastics—Cleveland Section, 7 papers. $2.50, members; $3.75, non-members. (See program, SPE Journal, August, 1959 issue, p. 745).

Plastics Finishing—Buffalo Section, 4 papers, $2.00, members; $3.00, non-members. (See program, SPE Journal, September, 1959 issue, p. 823).

Other 1959 RETEC Preprint Books

★ **Encapsulation, Printed Circuits, and Fluidized Bed Processes**—Northern Indiana Section, 15 of 23 papers presented. $3.00, members; $4.50, non-members. (See program SPE Journal, April, 1959, p. 319).

★ **Plastics in the Automotive Industry**—Detroit Section, 4 papers, $2.00, members; $3.00 non-members. (See program SPE Journal, May, 1959, p. 443).

★ **A Designer's Look at Reinforced Plastics** from the Pleasure Boat, Aircraft and Missile Industries Viewpoint—North Texas Section, 6 papers. $2.00, members; $3.00, non-members. (See program SPE Journal, April, 1959, p. 324).

★ **Plastics in the Metals Industries**—Pittsburgh Section, 10 papers. $3.00, members; $4.50, non-members. (See program SPE Journal, April, 1959, p. 324).

SPE PLASTICS ENGINEERING SERIES

Vol. II—Processing of Thermoplastic Materials, 705 pages. $14.40, members; $18.00, non-members.

Vol. I—Quality Control for Plastics Engineers. $3.98, members; $4.95, non-members.

ANTEC PREPRINT BOOKS

Vol. V, 1959, New York City, 96 papers. $7.50, members; $12.50, non-members. Single preprints of individual papers, Vol. V, while they last, $0.25 each, members; $0.40 each, non-members.

Vol. IV, 1958, Detroit, 94 papers. $5.00, members; $7.50, non-members.

Vol. III, 1957, St. Louis, 60 papers. $5.00, members; $7.50, non-members.

SPI individual members are entitled to SPE members' prices under a reciprocal agreement.

Books will be mailed postpaid if money is enclosed. Please address orders to:

Society of Plastics Engineers, Inc.
65 Prospect Street
Stamford, Conn.

Book Reviews

"Asbestos: Its Industrial Applications"

D. V. Rosato, Reinhold Publishing Corp., New York, 1959, price $5.75. 214 pages including Bibliography

This volume covers the use of asbestos in industry as well as presenting data on its various properties and the manufacturing methods utilized in the application of asbestos to industry. The concise but complete presentation covers the production of asbestos from the mine through the various industrial products. It shows the types of asbestos available from the various sources in the world as well as the properties that can be expected from the asbestos.

Industrially it covers the utilization of asbestos in cement products, tile products, as an insulation for heat, in friction applications, electrical usage, in textiles, in plastics, and in the many other applications for this versatile material. The book is completely annotated with suitable tables of physical and chemical properties of asbestos and the various forms in which it is utilized and combined. This book is recommended to the plastics engineer requiring a ready source of information on properties and uses of asbestos as a reinforcement for various plastics and resins. It also supplies an extensive bibliography to pursue areas where more precise information is needed.

Robert Steinman, Ph.D.
Garan Chemical Corp.

Source Book of Industrial Solvents

Volume 3: Monohydric Alcohols. Ibert Mellan, Reinhold Publishing Company, New York, 1959. 276 pp., $10.00.

This book is the third of a series of source books—for industrial solvents. In its treatment of monohydric alcohols, it is concise and remarkably full of pertinent information on manufacturing methods, properties, and uses of commercially significant products. Azeotropic mixtures of these alcohols with one another, or with other solvents, are listed, and their properties noted. Brief accounts of the historical development of the more important alcohols are given, along with relevant economic data. The graphic representations of the production flowsheets are clear and effective. The arrangement of the book makes it quite easy to find information about a particular alcohol; but somewhat more difficult, if one is seeking a solvent for a particular solute. Included in this book are three pages on not-so-alcoholic phenol—and unlooked-for bonus.

Charles H. Fuchsman
Ferro Chemical Corp.

Radioisotopes For Industry

Robert S. Rochlin and Warner W. Schultz, Reinhold Publishing Corp., N. Y., 190 pages, $4.75.

Radioisotopes for Industry is the newest entry in Reinhold's series of Pilot Books. These small books, which can be slipped into a coat pocket (5¼ x 5¼ x 6⅞"), are outstandingly useful in providing an authoritative, up-to-date and easily readable summary of the main ideas and uses for the subject under consideration. They provide a quickly read survey together with a list of sources of more detailed information, so that the reader does not get lost in a maze of thick volumes of difficult and highly technical material. In providing a good summary of a field, the Pilot Book series renders a notable service.

Radioisotopes for Industry—"purpose is to show engineers and plant managers how valuable these new methods can be in their own work". A very brief introduction is followed by chapters on well developed uses of radioisotopes in measuring and monitoring thickness of materials (of particular interest to producers of plastic film and sheeting) on radiography and autoradiography, and wear measurements (of possible interest to machine or mold makers). This is followed by a discussion of the use of radioisotopes in manufacturing, finding leaks with radioisotopes and use of tracers in research and development. The final three chapters deal with equipment, training, protection, and the projected future of radioisotopes.

It should be stressed that this book is for the engineer or executive and does not assume any prior knowledge of the subject. *Dr. Jesse N. Day*

(Continued from page 899)

4:45—Blow Molded Plastic Containers,

G. S. Brown, Plax Corporation, Hartford, Conn.

The plastic bottle has become accepted and in many instances a necessary means for containing and dispensing a large variety of liquids and powders. A review of the various types of blow molded plastic containers will be given to elaborate upon the obtainable functional properties. Design considerations in the development of a plastic container will also be presented along with manufacturing processes of blow molding, methods of decoration and labeling, and selection of raw material for end use applications.

SECTION B

Ned L. Roberts, Moderator

Steven Enterprises, Palo Alto, Calif.

3:45—Cook-In Pouches,

Fred B. Shaw, Flexible Packaging Div., Continental Can Co., Mt. Vernon, Ohio

The cook-in pouch has been under development for two or three years but is only now beginning to effectively enter the market. This pouch generally contains an individual serving of a prepared food and is sold in the frozen condition. The entire package is inserted in water and brought to a boil for perhaps ten minutes, after which time the pouch is removed from the boiling water and the contents served on the consumer's individual plate. Such pouches must withstand both freezing and boiling without delamination or becoming unsealed and they must resist various food components. They must retain flavor and therefore have low WVTR and low O_2 permeability properties.

4:15—Packaging with Linear Polyethylene,

R. Vernon Jones, Phillips Chemical Co., Bartlesville, Okla.

Linear polyethylene is finding a host of applications in the packaging industry. The strength, stiffness, chemical inertness and barrier properties of the linear polyethylenes lend them to light weight, low cost, efficient packages. The discussion will include examples and comments on injection and blow molded bottles and jars, vacuum formed food containers, transparent overwrap films, and a new heavy-wall industrial bag.

4:45—Molded Expanded Polystyrene Packaging

Joe Slapnick and E. Y. Wolford, Koppers Co., Los Angeles and Pittsburgh, Pa.

Molded expandable polystyrene packages offer high strength, light weight, low MVT, negligible water absorption, neutral pH, thermal insulation, attractive appearance and high energy absorption. New molding techniques plus low raw material costs have produced consumer, military and industrial packages less expensive than conventional materials for typewriters, baby gift sets, candy, comb and brush sets, electronic tubes and missile components. The recent development of formable sheet and resilient polystyrene foam will expand this field further.

About Members

L. P. Thies was appointed manager of the Polyester Products Dept. of Goodyear, while **W. E. Kelly,** former manager of the division's St. Louis office was named to head the new Adhesive Dept. These are new sales departments established within the Chemical Division of The Goodyear Tire & Rubber Co. Mr. Thies, an affiliate of the Detroit Section of SPE, joined Goodyear in 1956. He was graduated from Kent State University with a B.S. degree in chemistry and holds a master's degree in business administration from Indiana University.

Mr. Kelly has been with Goodyear since 1949 and had previous experience in textile and adhesive sales. He was assigned to the Chemical Division's New York office and in 1954 was transferred to the St. Louis office as district manager. A member of the Kentuckiana Section of SPE, Mr. Kelly is a graduate of Hofstra College, Hempstead, N. Y., with an AB degree and received a master of science degree from Lehigh University.

George Dingman has been appointed general manager of Chippewa Plastics Co., a division of Rexall Drug and Chemical Co., and a producer of polyethylene film. In his new position, Mr. Dingman will assume administrative responsibilities for all operations. He joined Chippewa plastics in 1955 and most recently was director of purchases and planning. Prior to that he was engaged in pharmaceutical sales with the Smith-Dorsey Division of Wonder Co. A graduate of the University of Nebraska, he is a member of the National Association of purchasing agents and the Upper Midwest Section of SPE.

Edwin E. Kasha has been named head of the Color Development Laboratories for Foster Grant Co., Inc., of Leominster, Mass. Mr. Kasha formerly was chief colorist for Koppers Co. He is a member of the Pittsburgh Section of SPE, the Pittsburgh Paint & Varnish Production Club and the American Institute of Chem-

W. E. Kelly

L. P. Thies

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ical Engineers. Mr. Kasha received his Bachelor of Science degree in chemical engineering from the University of Pittsburgh.

John J. Reed has been appointed manager of Isocyanates Market Development, Allied Chemical's National Aniline Division. Mr. Reed joined the National Aniline sales staff in 1954. He is a graduate of Villanova University and received a B.S. degree in chemical engineering in 1950. Mr. Reed is a member of the Newark Section of the SPE.

Philip I. Johnson was promoted to product manager suspensions resins, in Diamond Alkali Company's plastics division. Succeeding Mr. Johnson in his vacated post as plastics division sales representative in the New York-Wilmington area is **Peter W. Rizzo,** formerly associated with the National Lead Co., Brooklyn, N. Y. Mr. Johnson joined the Diamond organization in June, 1958 as a sales promotion representative with 11 years previous experience in the plastics field. In this newly created post at the company's headquarters offices, Cleveland, he will assume responsibility for market planning, sales promotion and advertising of suspension resins. He is a graduate of the University of Wisconsin, Madison, Wisc., where he earned his B.S. degree in chemical engineering. He is a member of the AGS, and the New York Section of SPE.

Mr. Rizzo was a sales development representative for various polyvinyl chloride stabilizers for National Lead before joining Diamond. He holds a M.S. degree in organic chemistry from the University of Chicago, and a B.S. degree in chemistry from Harvard University. Mr. Rizzo is a member of the New York Section of SPE.

John A. Parsons and **J. William Arpin,** both members of the Binghamton Section of SPE have been elected vice presidents by American Plastics Corp., a subsidiary of Heyden Newport Chemical Corp., Mr. Parsons, named executive vice president of American Plastics, has served as director and vice president in charge of production. His career in various plastics production and management capacities spans more than four decades in the plastics industry. Mr. Arpin, formerly assistant plant manager, has been appointed vice president for engineering and deveolpment. He is a specialist in the field of injection molding, mold and tool design, and machine design with numerous patent credits. Mr. Arpin is an alumnus of the University of Florida, and is chairman of the Binghamton Section membership committee of SPE.

Willard deCamp Crater, Jr., has been appointed executive vice president and general manager of the American Molding Powder & Chemical Co. Mr. Crater, who is affiliated with the Newark Section of SPE, received his education at the University of Delaware and at Rutgers University, and was formerly employed by W. R. Grace & Co. He recently completed a round the world business trip promoting plastics.

Willard deCamp Crater, Jr.

George R. Smoluk

George R. Smoluk has been appointed engineering editor of Modern Plastics magazine, according to an announcement by Hiram C. McCann, editor. Mr. Smoluk is a graduate of Princeton University with the degree of Master of Science in engineering, specialist in plastics. In addition to four years as new product engineer at the Bakelite Co. division of Union Carbide, Mr. Smoluk has served as a planning engineer with Celanese Corporation, as a field engineer with Du Pont, and plastics application engineer with General Electric. He is a member of the Newark Section of SPE.

Dr. Kurt C. Frisch, a member of the Detroit Section of SPE, was appointed director of polymer research at Wyandotte Chemicals Corp., it was announced recently.

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Section News

Baltimore-Washington

Blow-Molding Discussed

TAYLOR E. BRICKHEAD

One of the fastest-growing areas of the plastics industry was the subject of a panel discussion held September 8, 1959—blow molding. Moderator and first speaker was Vernon Hill of the Continental Can Corp. The program covered economics, equipment, applications, and materials of and for blow molding.

Later this month, on October 20, the Baltimore-Washington Section will hear Professor Albert G. H. Dietz of M.I.T. speak on the Moscow Fair and specifically about the latest developments in the plastics field.

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Chicago

Scholarships Awarded

HARRY E. BENNETT

The Chicago Section of SPE has selected Alex John Bart, of Chicago, to receive their $2000 scholarship award, offered to engineering students entering college this Fall. Alex ranked third in his class of 655 at the Lane Technical High School, Chicago. He was one of 2,513 out of 45,548 National Honor Society members who scored 142 or higher to qualify in the 1959 National Honor Society scholarship finals. Alex will major in chemistry at Northwestern University.

Monterrey, Mexico

Polystyrene Processes

JOHN P. BRECKER

The second regular meeting of this new section was held on August 17, 1959 at the Restaurant "Los Acros." Guest speaker was Alfredo Corella, General Manager of "Aislantes Leon", S.A. His subject was The Development of the Processes Used for Manufacturing Foamed Polystyrene."

Newark

Injection Molding vs. Vacuum Forming

DAVID L. SAHUD

A panel discussion on Injection Molding versus Vacuum Forming was held at the September meeting. Ernest E. Moslo, President of Moslo Machinery Co., Cleveland, Ohio, was panel moderator. Other panel members were John R. Kent, Monsanto Chemical Co., Robert E. Kostur, Comet Industries, Frank L. Quinby, W. R. Grace & Co., and Edward Rowan, Parkway Plastics.

Rochester

New Plastic

JOHN T. BENT

Dr. Harry Gates, Du Pont, was the speaker at the September meeting. His subject was Du Pont's new acetal resin, Delrin. Dr. Gates was directly involved with the development of this new resin, from the initial research six years ago to current production phases.

Central New York

Extrudable Teflon

JAMES R. LAMPMAN

The Central New York section kicked off their new season with a dinner-meeting held September 10, 1959. Speaker for the meeting was William N. Brinker, Du Pont. He spoke on Teflon resins and particularly their new melt extrudable polymer, Teflon FEP.

Employment & Service Guide

POSITIONS OPEN

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Box 68, Washington, West Virginia

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G. H. Share, Salary Administrator
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Exceptionally qualified for Research and Development in problems of plastics materials and systems using plastics, including material selection, processing techniques and enduse and reliability testing. Eleven years experience including six as supervisor of plastics molding and testing laboratory with record of proven accomplishment in the solution of unusal problems. Experience encompasses injection, transfer and compression molding, vacuum forming and fabrication of most materials, especially the polyolefins, fluorocarbons, diallyl phthalate and epoxy resins. Desire supervisory position with a progressive company which will provide room for professional growth and advancement. Age 34 with B.E. in chemical engineering and graduate work in organic chemistry. For detailed resume, reply Box 5859, SPE Journal, 65 Prospect St., Stamford, Conn.

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SOCIETY OF PLASTICS ENGINEERS, INC.
January 12, 13, 14 and 15, 1960
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